Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

OCTOBER 1950

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wet-strengthen your paper with the UFORMITE resins

All over America, *housewives* pass judgment on your frozen food wraps. Lack of wet-strength and pick-resistance can bring a barrage of complaints—and rejections.

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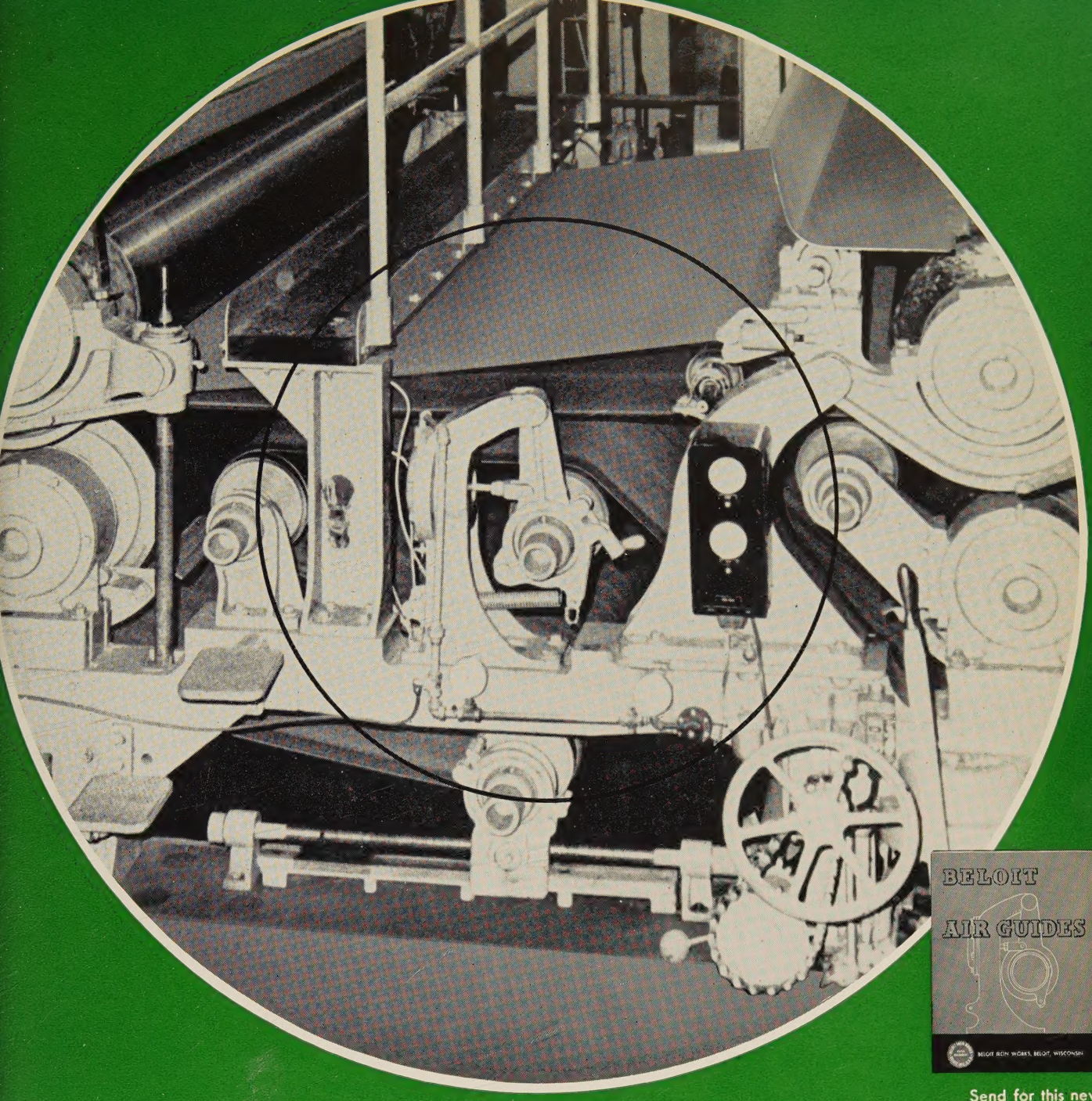

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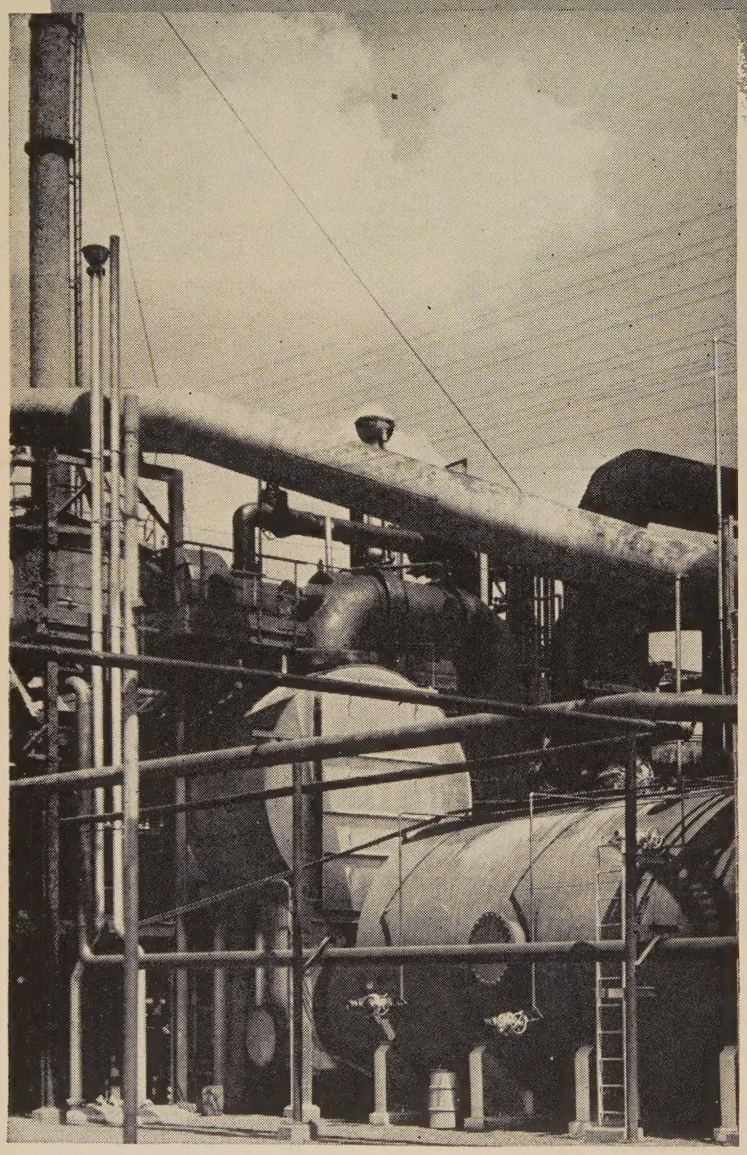

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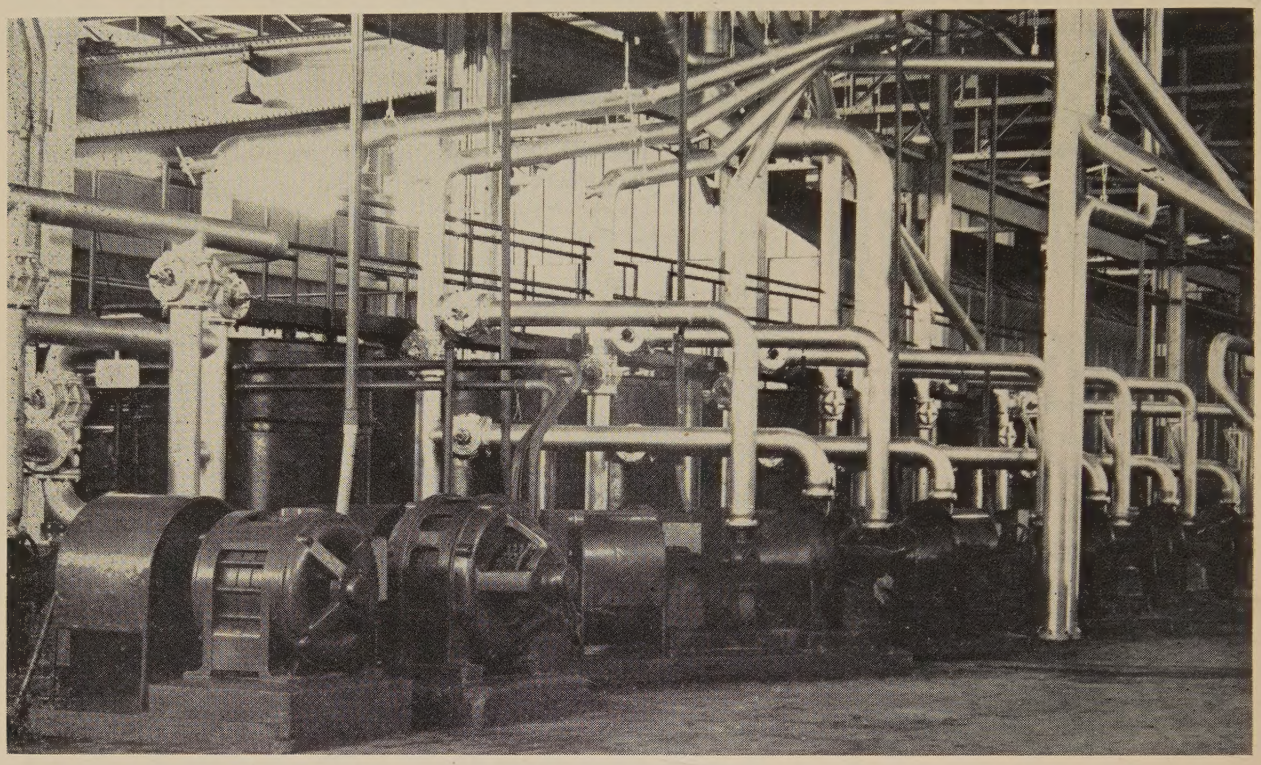

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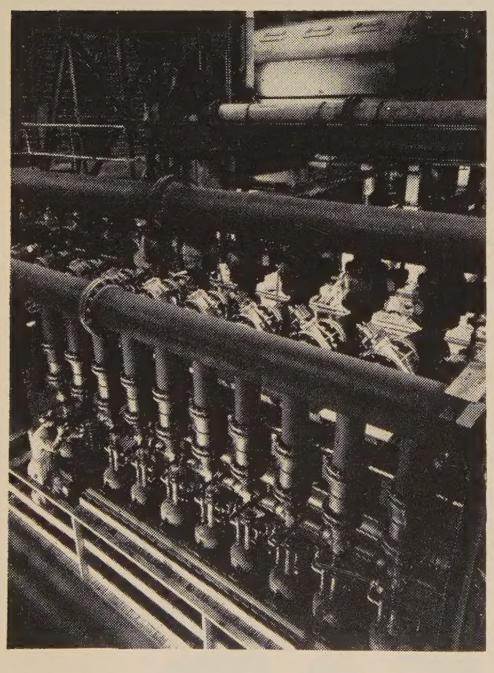

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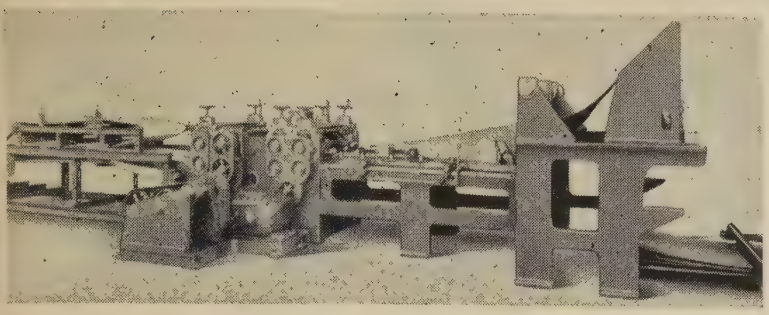

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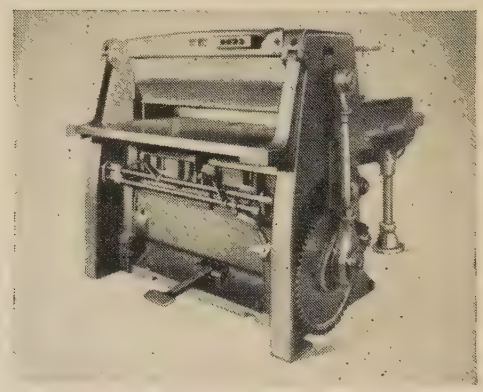

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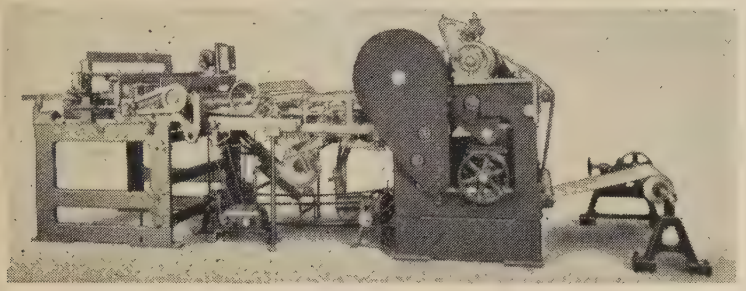

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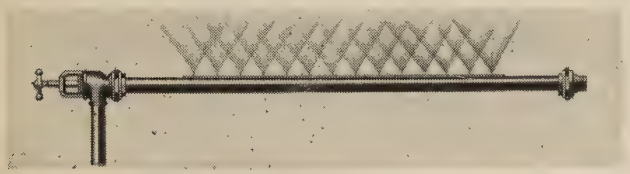

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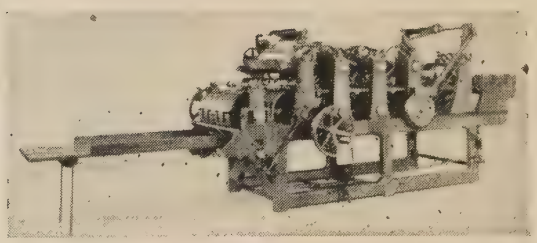

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S & W Bottomers are designed for high production and minimum spoilage with many new features for lowering bag production costs. Bottomers are made in several sizes. Produce standard satchel or narrow valve bottom.

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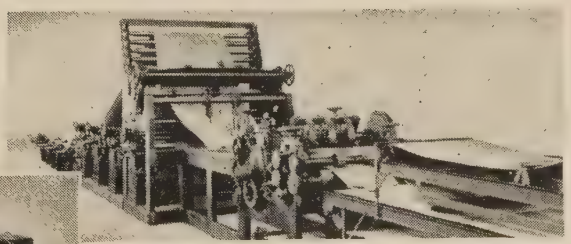

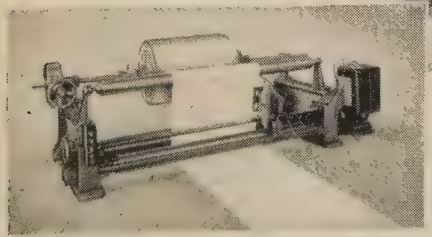

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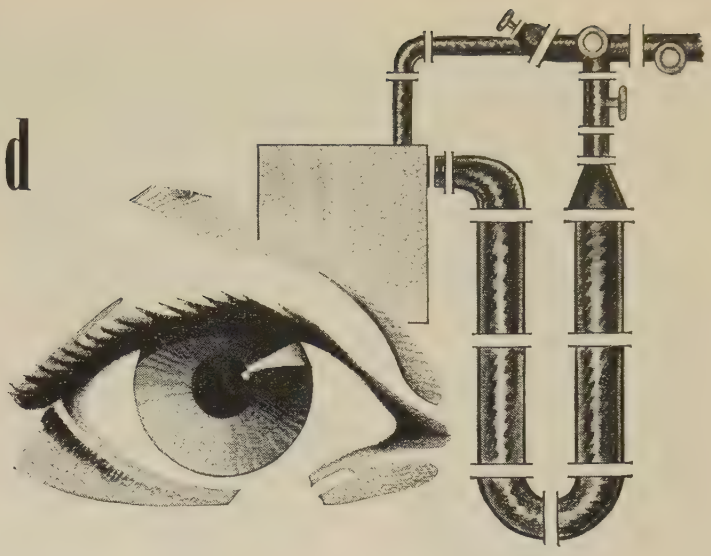

Visual inspection of the chlorination stage through sight glasses in the stock pipe leading from the chlorinator and another after the bleacher is approved practice today. But back in 1931 when the first Hooker continuous chlorination system was designed to overcome the disadvantages of a batch process, no provisions for visual inspection of the stock were included. Redesigning the equipment to provide for adequate sight glasses was another development made cooperatively by engineers of the pulp and paper industry and the Hooker Technical Staff.

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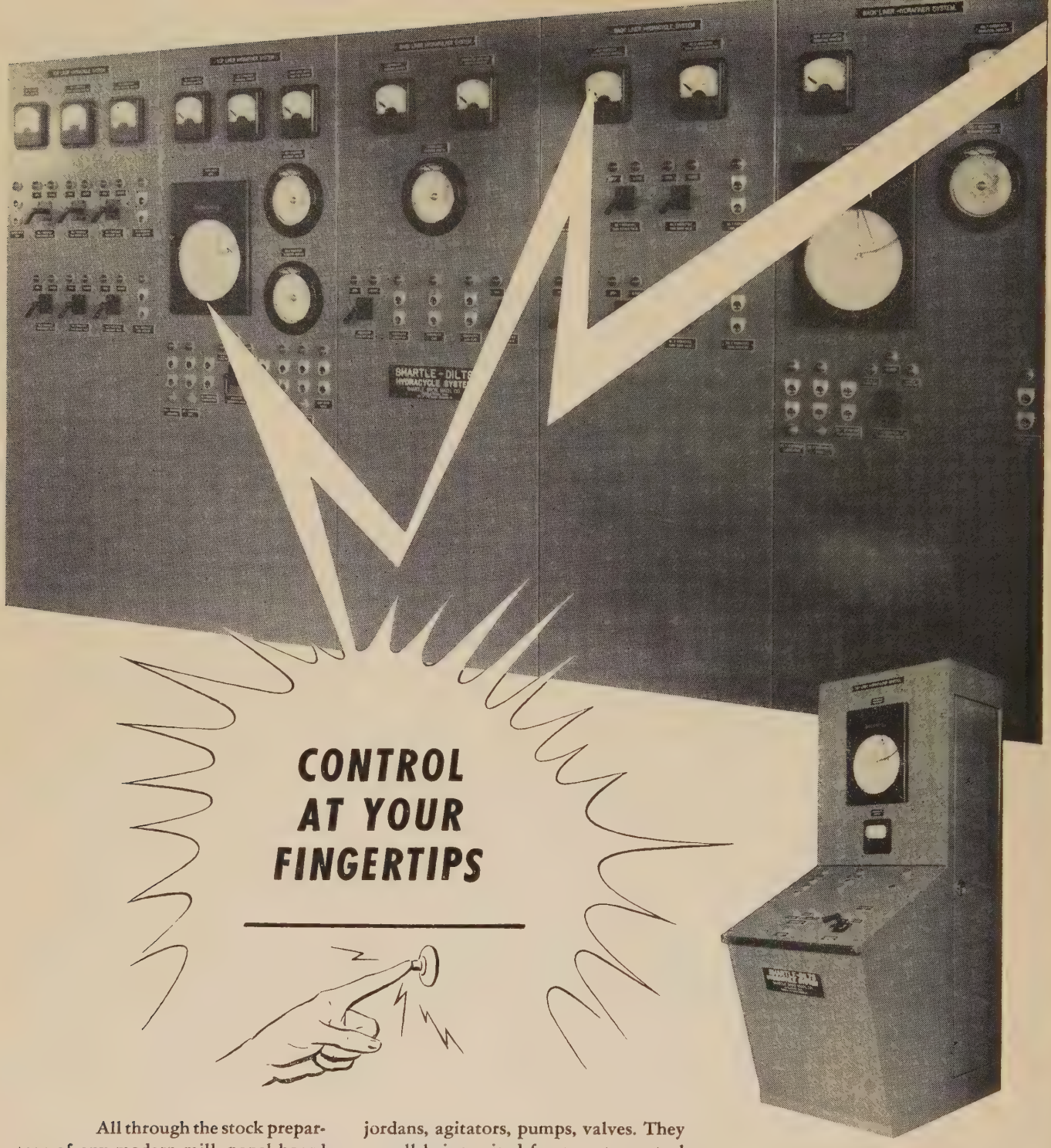

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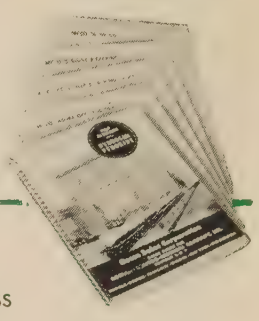

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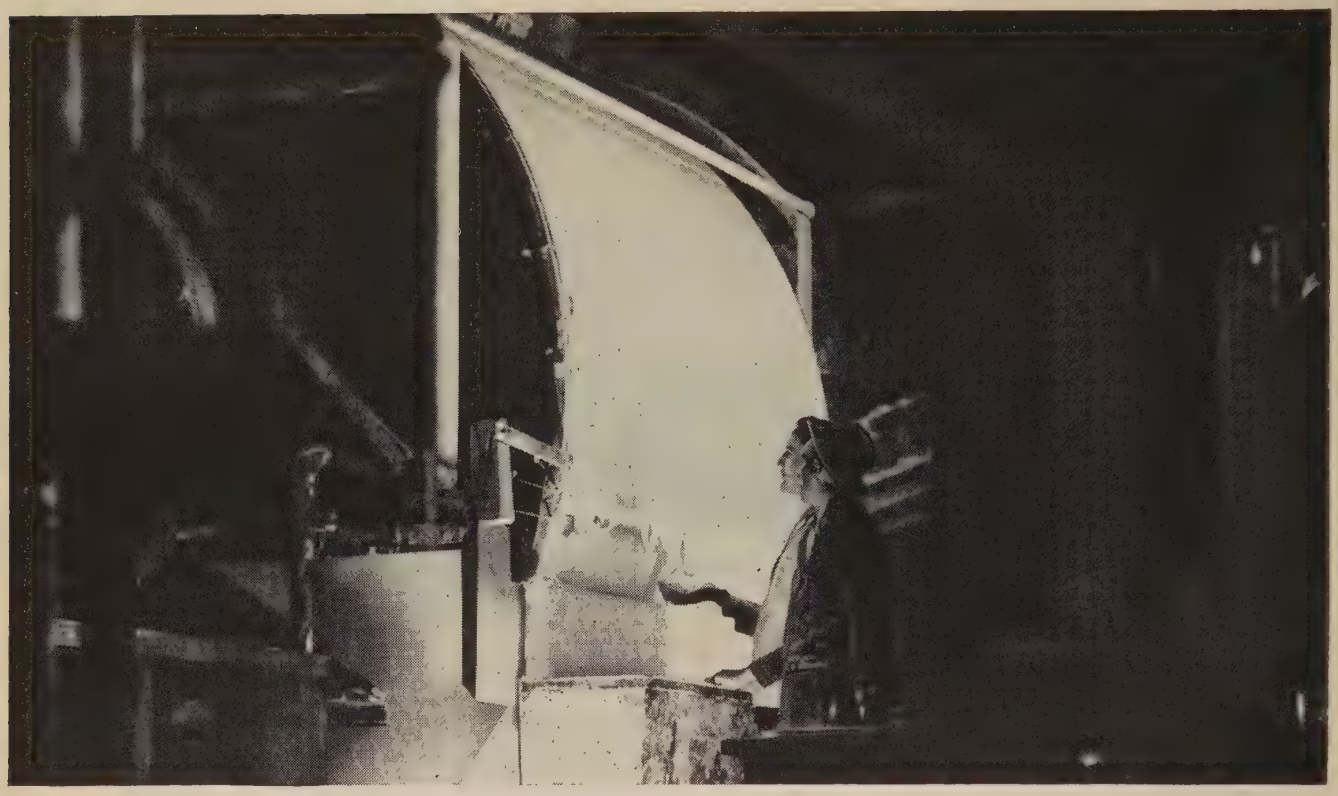

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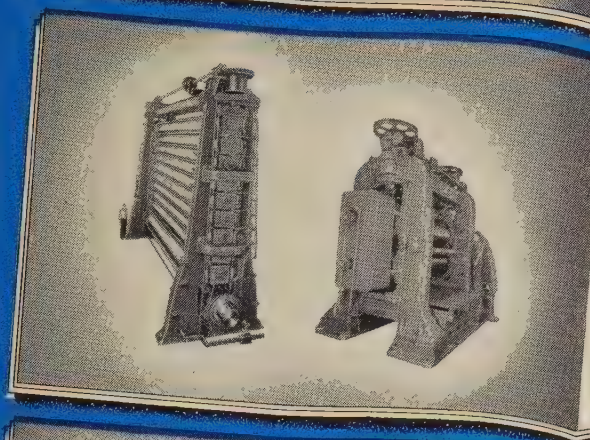

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Farrel paper-machine calenders can be made with rolls in any face length up to 312".

Housings are box section type, possessing great stiffness and rigidity. Roll journals are mounted in either sleeve-type or antifriction bearings. For raising the rolls, calenders are equipped with electric, hydraulic or ratchet lift, all operated from the floor.

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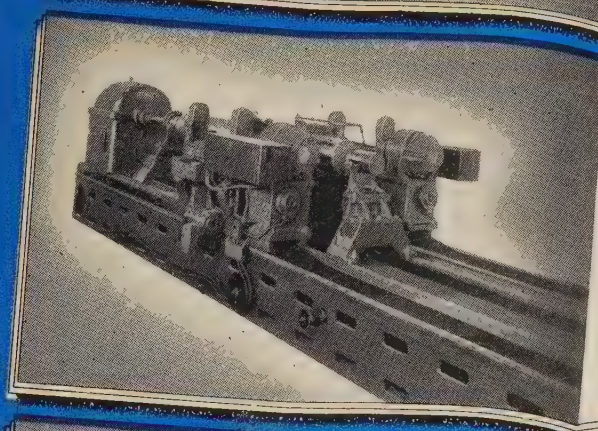

ROLL GRINDERS

Farrel two-wheel, swing rest roll grinders, designed especially for grinding the long rolls used in the paper industry, are capable of producing unusual accuracy and smooth finish. The exclusive swing-rest principle, with the use of two grinding wheels, reduces to a minimum the minor inaccuracies imparted to the roll when grinding with any fixed-rest machine.

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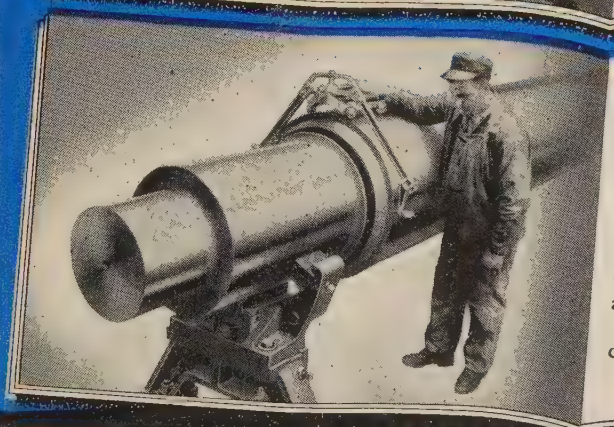

ROLL CALIPERS

Farrel roll calipers provide the quickest method of checking accuracy of roll diameter and crown, either when the roll is in or out of the grinder. They will detect variations in roll diameters down to .0001". Readings can be taken at any point along the face of the roll, and at any number of points. Rolls can be checked in a fraction of the time required to test them with a micrometer or steel tape, and with far greater accuracy.

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Kraft Papermaking

FREDERICK G. SAWYER, CHESTER T. BEALS and ARTHUR W. NEUBAUER

THE STORY OF paper is romantic and exciting. Its early history tells of Chinese scholars, an emperor, Arab conquerors, and merchant caravans. In the Seventeenth Century, the art of papermaking came to America.

In 1798, a French workman named Louis Robert announced that he had discovered a way to make, with one man and without fire, by means of machines, sheets of paper of a very large size, even 12 feet wide and 50 feet long. Robert secured a patent on his machine but did not have the money to finance it. Two wealthy London stationers, Henry and Sealy Fourdrinier, became interested in the proposition. In 1804, the first successful machine was started at Frogmore, England. The paper machine was named after the Fourdriniers because of their persistent faith which, however, led them to bankruptcy. After having spent £60,000 and being reduced to penury, they petitioned Parliament for compensation. Fortunately, their labors were appreciated and £7000 were voted them. Although their machine has been changed somewhat in details, it is still fundamentally the same as the one originally invented (*6*).

Now that mechanical means for making paper had been developed, it was soon realized that rags and straw were not available in sufficient quantities to sate the machine's voracious appetite. A new fibrous material had to be found. In 1850 a German named Frederic Kellar studied the pulpmaking methods of the wasp, and with a mechanic named Henry Volter devised a machine for grinding wood into fibers. Two years later, Hugh Burgess, an English inventor, was issued patents for pulping by what is now known as the soda process. Man was improving on the wasp.

In 1865 American chemist C. B. Tilghman discovered the method known today as the sulphite process for cooking wood. In 1884 the kraft process was developed. Because of the great strength of kraft pulp, its production has greatly increased in recent years particularly in the southern states and on the Pacific Coast. This process, as applied in the Camas, Wash., mill of the Crown Zellerbach Corp., was described in a previous staff-industry collaborative report (*14*). The present article is a sequel describing how this same mill makes paper from kraft pulp.

Modern papermaking can be considered a chemical and engineering process. The importance of chemicals is emphasized by the fact that more than 600 are now used to improve pulp and paper quality and to build new paper characteristics. The pulp and paper industry is the third largest consumer of chemicals, following only fertilizer and rayon (*15*). The basic pulping chemicals used include caustic soda, lime, salt cake, soda ash, sodium sulphide, and sulphur. In the last 20 years, the use of these chemicals in pulp manufacture has increased at a rapid rate. For example, use of caustic soda has increased more than four times; chlorine has increased three times; soda ash almost three times; and salt cake seven times (*13*).

After the pulp is made, many more chemicals are needed to make it into a sheet of paper. These include alum, colors, starches, proteins, resins, and pigments such as clay, talc, titanium dioxide, diatomaceous earth, alkaline carbonates, and calcium and barium sulphates. At least 200 chemicals can be used for coating paper. Chemicals are also used for deinking paper and for making the sheet strong when wet. World War II showed dramatically that wet-strength papers are here to stay. Chemical techniques have been applied to the improvement of paper packaging, including development of multiwall bags, resin-impregnated papers, resin coatings, waterproof adhesives, and special sealing compounds for bag closures.

CAMAS, WASH., MILL OF CROWN ZELLERBACH CORP.

Today we have all the necessary components to make kraft paper: the kraft pulp, the fourdrinier machine, good water, chemicals, and skilled American man power. These factors are combined efficiently in the Crown Zellerbach Corp. mill at Camas, Wash. Some idea of the Camas mill's size can be gained from its annual materials requirements shown in Table I. Although this plant makes a wide variety of pulps and papers, only the manufacture of kraft bag, wrapping, and specialty grades, such as waxing, food cartons, meat wraps, etc., will be discussed in detail. A summary of the general operations at Camas follows:

Table I. Camas Mill Annual Requirements

Wood, board ft.	182,000,000	Lime rock (and dolomite), lb.	36,529,000
Water, gal.	23,785,800,000		
Electricity, kw.-hr.	257,325,000	Salt cake, lb.	11,860,000
Lubricating oil, gal.	71,500	Alum, lb.	5,957,000
Grease, lb.	36,500	Lime, lb.	10,107,000
Steam, lb.	3,165,119,000	Rosin size, lb.	4,773,000
Hog fuel, cu. ft.	34,358,000	Pigments (clay, talc, titanium dioxide), lb.	4,587,000
Chlorine, lb.	16,554,000		
Fuel oil, barrels	210,300	Dyestuffs, lb.	476,000
Sulphur, lb.	30,788,000		

After being prepared (*14*), the kraft pulp or stock is stored at 17% consistency. When needed for papermaking, the pulp is diluted, screened, thickened (deckered), and sent either to the bleach plant for further processing or to the beaters.

A multistage process is necessary for bleaching kraft pulp in order to obtain the required degree of whiteness

FREDERICK G. SAWYER, Associate Editor Industrial Engineering Chemistry, CHESTER T. BEALS and ARTHUR W. NEUBAUER, Crown Zellerbach Corp., Camas, Wash.

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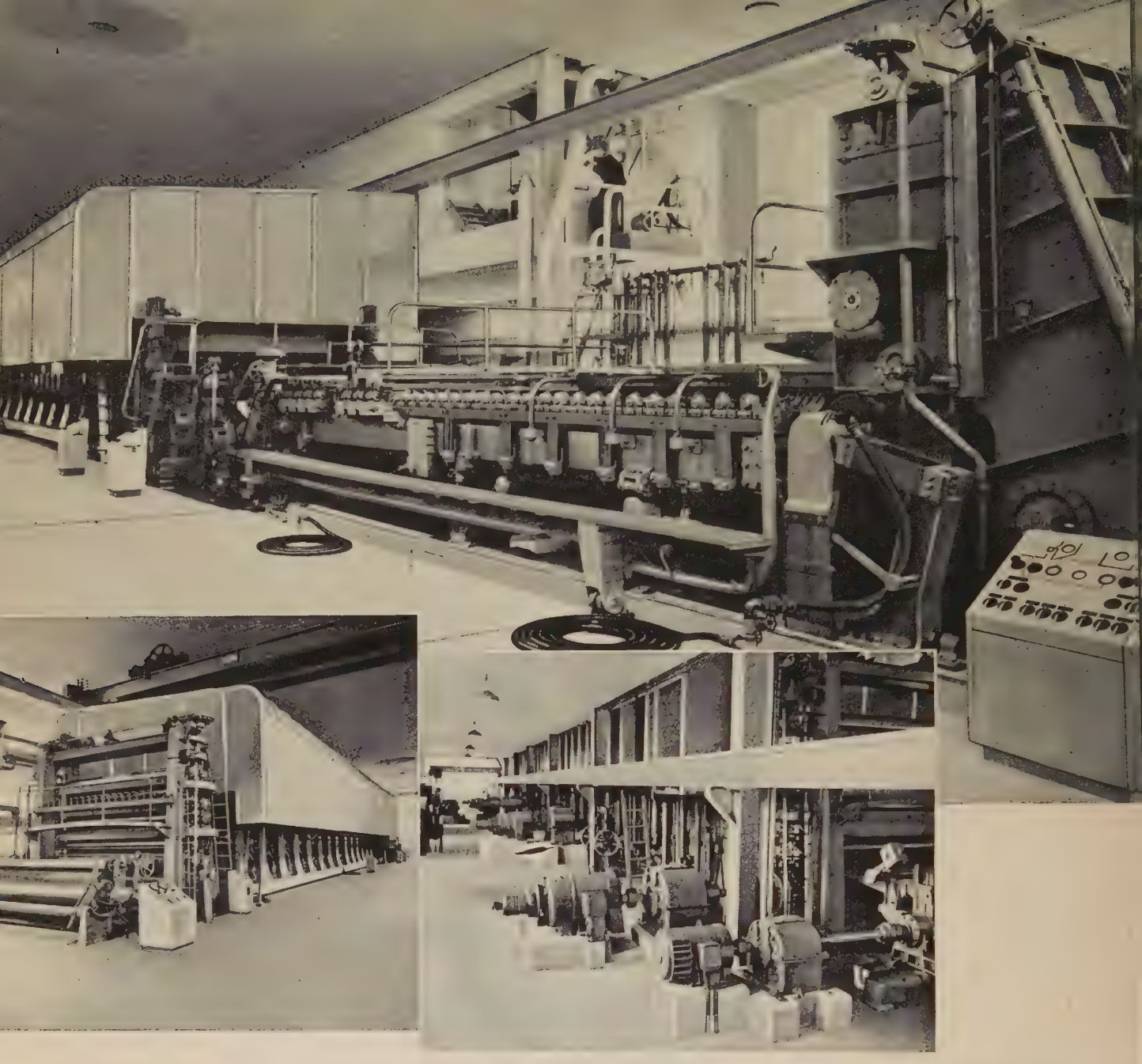


FEATURES OF THE 138" PUSEYJONES MACHINE AT PAPIERFABRIK UTZENSTORF

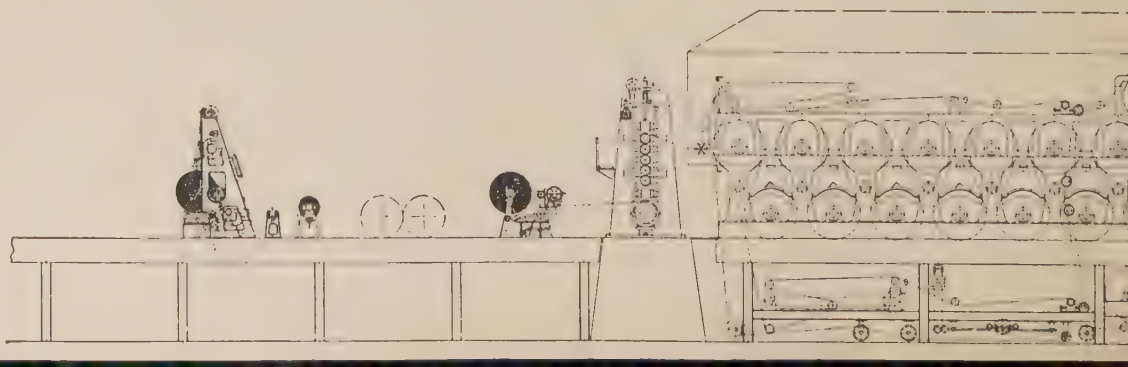
Improved type Flow Spreader combined with 6 ft. high adjustable slice. Special stock level control.

Fourdrinier Part suitable for wire 75 ft. long and 138" wide, seven 7" wide monel suction boxes with oscillating device, 30" suction couch roll with lump breaker roll and pneumatic draw roll; "Rapi-drape" wire changing device and adjustable wire pitch arrangement.

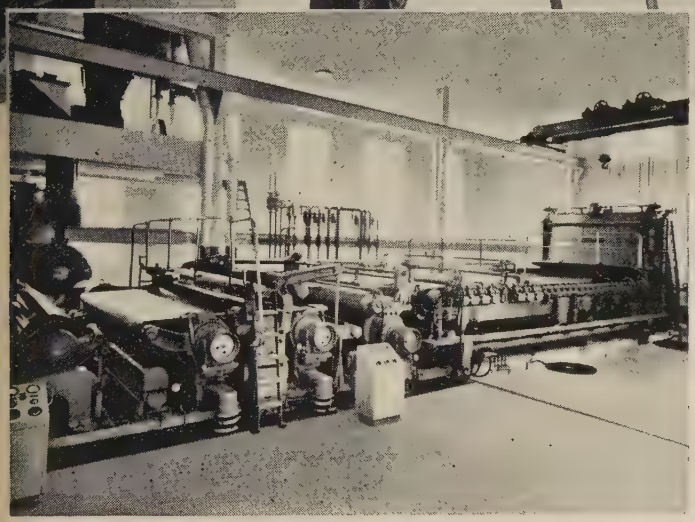
Press Part of dual press arrangement with two 26" rubber covered suction rolls and 32" Stonite covered center roll, also 18" rubber covered top press roll, pneumatic nip loading; complete felt equipment and Vickery conditioner.

One set of smoothing rolls located between press and dry parts, equipped with 24" upper rubber covered roll and 26" bronze cased lower roll, pneumatic nip loading.

Dry Part in two main sections having one upper and one lower felt in each section; first section comprising 16—60" paper and 4—60" felt dryers; second section comprising 16—60" paper and 2—60" felt dryers; improved type enclosed gearing; Bowser lubricating system; rope feed arrangement with automatic transfer



Line drawing of the new Puseyjones Fourdrinier Machine at Papierfabrik Utzenstorf, Switzerland. Designed and constructed for a maximum operating speed of 1200 feet per minute with Brown Bovari sectional electric motor drive. All rolls except bottom calender equipped with anti-friction bearings.



Puseyjones Crosses the Swiss Alps

This Puseyjones Fourdrinier Machine is now in operation at the modernized plant of Papierfabrik Utzenstorf, Switzerland. The addition of new equipment and complete improvement of existing facilities have made this Swiss mill one of the most modern and outstanding on the European continent.

From the improved type Flow Spreader to the 2-drum Winder, this new Puseyjones Machine incorporates the latest features in paper-making machine design and construction. It is producing news and directory paper at the rate of 75 tons daily.

Puseyjones Machines are in operation in the leading mills across the country, and in many parts of the world, giving continuous, quality production at high speeds. If you are thinking of modernizing or adding to your present equipment, then get the benefits of Puseyjones experience. Puseyjones engineers will be glad to discuss your problem. Write us today.

between sections; automatic motor operated felt tension control devices; improved type steam joints with dual revolving siphons.

Calender Stack comprising 8 rolls with independent Bowser oiling system for all roll bearings; pneumatic nip loading.

Pope type Uniform Speed Reel of improved design, with pneumatic adjustable loading mechanism.

Unwinding stands equipped with water-cooled brake and pneumatic tension device.

Puseyjones Winder with hydraulic operated roll unloading mechanism.

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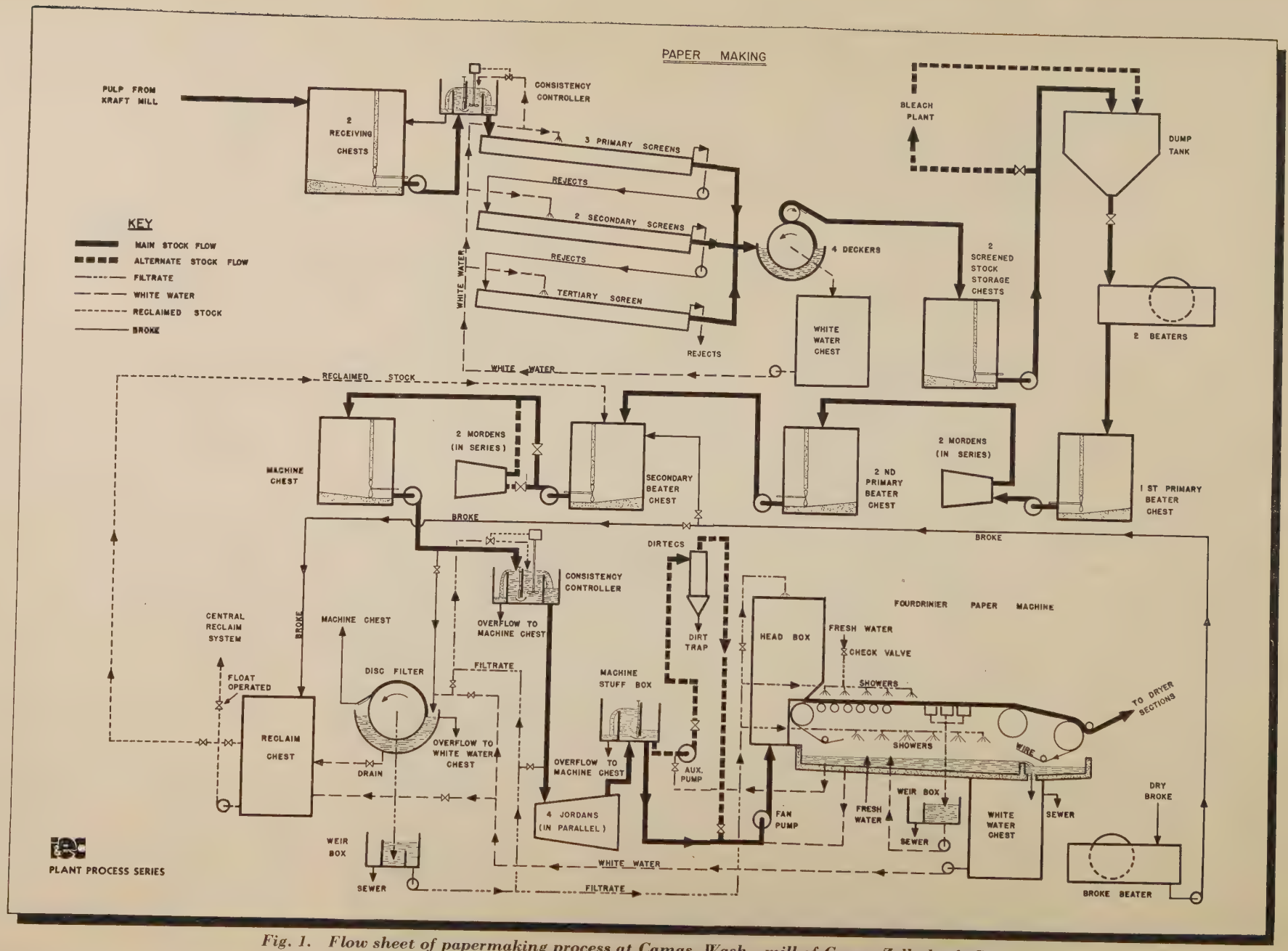

Fig. 1. Flow sheet of papermaking process at Camas, Wash., mill of Crown Zellerbach Corp.

GROWING A NEW "CROP" OF TIMBER EVERY 80 YEARS

This harvesting and re-growing area at Vail, Washington, reflects the long range planning for successive timber crops

THE WEYERHAEUSER forestry policy is based upon the growing, protecting and harvesting of trees . . . looking toward permanency and improved utilization of the forest crop. The photograph above illustrates this Weyerhaeuser principle of progressive harvesting and regrowing . . . a continuous cycle which will provide perpetual forest crops.

To grow a fir seedling into a tree of mature size requires from eighty to one hundred years. As a young tree progresses through the various stages of growth, it must be protected from fire, disease and insects until it is ready for harvesting.

Weyerhaeuser alone and in cooperation with others in the forest industry, now operates eleven certified Tree Farms. These areas, under modern forest management, will continue to produce new crops of timber as generations come and go. The Weyerhaeuser Timber Company grows trees because it is good business . . . the cycle of harvesting and growing providing a permanent source of raw material for the Pulp Division.

without weakening the fibers. Bleaching consists of removing lignin residues and the brown color. Chlorine is added and the subsequently formed insoluble chlorinated lignins are solublized with sodium hydroxide. The pulp, finally whitened by hypochlorite treatment, enters the beater room parallel to the unbleached pulps.

In the beater, the fibers are fibrillated (ends frayed) and bruised to develop strength characteristics in the subsequent paper. Some cutting also takes place. Chemicals such as colors, resins, size, and fillers are added.

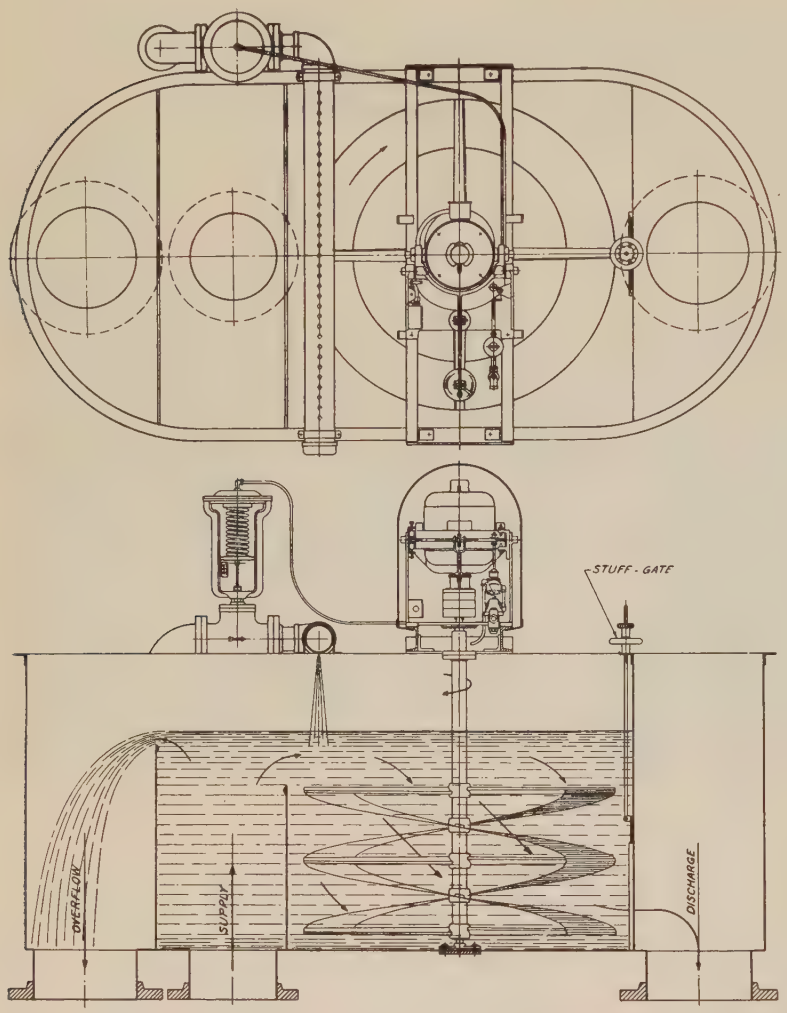

Fig. 2. Consistency controller

From the beaters, the pulp passes through a series of refiners (Mordens and jordans) where for the most part the fibers are shortened to give further desired sheet characteristics. The stock is then metered, diluted, and introduced to the paper machine. Here it is distributed uniformly across a continuously moving wire cloth which allows water to drain through. Table rolls, suction boxes, and suction rolls aid this natural drainage.

The wet web is transferred to an endless woolen blanket which conveys it between press rolls to remove more moisture. There are also rolls in the press section to control sheet thickness and surface. This is followed by drying over steam-heated drums where the sheet is held against the drums by a continuous canvas felt. In the drying section there are also smoothing rolls and application facilities for surface treating or sizing. The sheet passes from the last heating drum through a calender stack for final machine finishing. It is then reeled and rewound to be either cut for shipment or subjected to further finishing.

STORAGE AND SCREENING

The manner in which the many materials are combined and processed to produce a specialty grade paper will now be considered in detail, as applied in the Camas, Wash., mill of Crown Zellerbach. The kraft pulp is stored in two high consistency chests (85 tons of air-dry fiber) made of red tile-lined concrete (Fig. 1). The small amount of soda compounds (30 pounds per ton of pulp) in the pulp would eventually attack unlined concrete resulting in spalling. The tile lining ensures cleanliness of the stock and long life for the chests.

In order to remove the 17% consistency pulp for papermaking, water at 50 p.s.i. pressure shoots in toward the center of the chest from a number of jets at the bottom. The lowermost pulp is washed to the center of the chest and down through two centrifugal pumps. The stock, now diluted to 2.5 to 3% consistency, is pumped to two 12-ton tile-lined concrete receiving chests. A 36-inch propeller-type impeller agitates the stock by forcing it between two vertical walls which divided the chest into approximate thirds. The stock flows down the center channel; divides into two streams flowing behind the walls to the other end; then the streams converge and are forced through the impeller again. The splitting and converging of the two elliptical flows provide agitation. Flexibility of operation can be obtained by varying the pitch of the impeller blades. The same size impeller can be used in other chests with only an adjustment of the blade pitch to take care of consistency differences.

The stock is then pumped to a consistency controller (*7E*) where it is diluted to 1.5 to 2% (Fig. 2). An over-flow line returns excess stock to the receiving chest. In the consistency controller there is a central helical ribbon 30 inches in diameter and 8 inches wide. As the agitator is rotated to 50 r.p.m. by a 1.5-hp. motor, the upward reaction is related to the consistency at that moment. An increase in consistency produces greater upward thrust on the agitator, raising the unit, and thereby changing position of the pilot valve stem. This in turn opens the dilution valve to admit more water. A decrease in consistency causes the unit to lower, admitting less water. The controller cannot thicken but can dilute the incoming stock. The mechanism will control consistency within approximately 0.3% on either side of the setting.

The important removal of shives and dirt is accomplished in screens. The pulp flows to a head box where it is diluted to 0.6% (Fig. 3). It subsequently flows to

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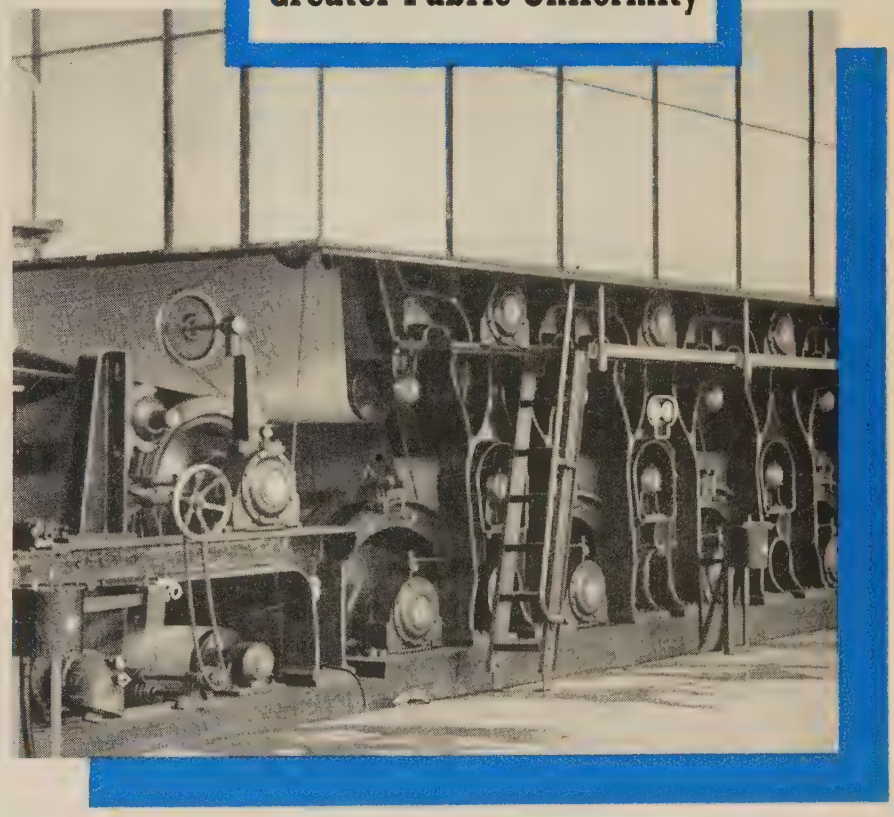

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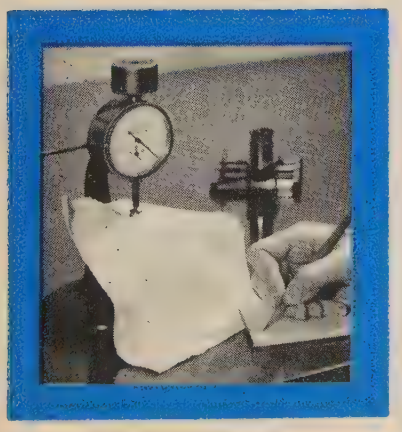

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three rows of primary screens. Each row comprises three 14-plate screens pitched 0.5 inch to the foot. Rubber diaphragms under the screens are activated by four-point cams. The plates are made of chromium-plated bronze, 43 inches wide and $11^3/_8$ inches long, having narrow slots six to the inch with an opening of 0.012 inch (known as 12-cut). Unplated bronze wears away too quickly and does not retain the slot shape. The screen plates are mounted on brass containers and are held in place with fasteners that can be easily adjusted and removed for inspection and cleaning.

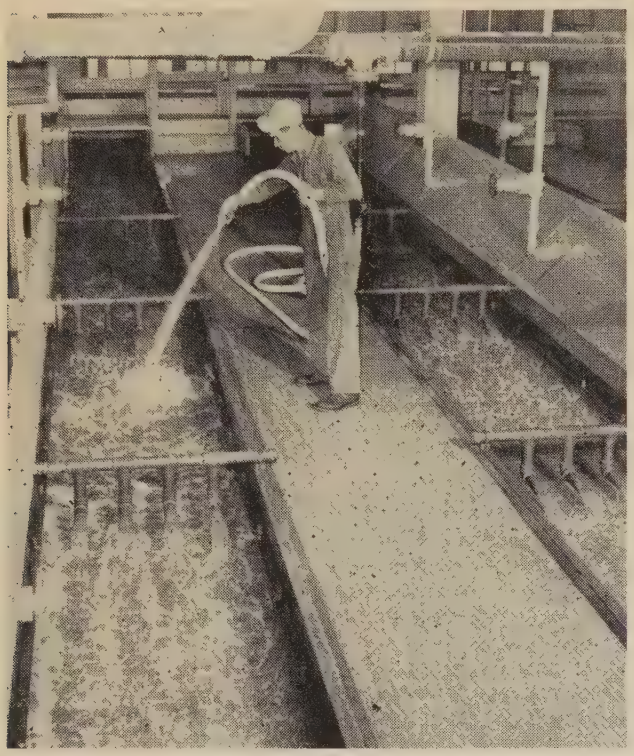

Fig. 3. Pulp running through primary screens

The rejects (pulp, shives, and dirt) that do not pass through the slots of the primary screen are diluted to 0.3% consistency and pumped to the head box feeding two rows of secondary screens. These are similar to the primary screens but the slot openings are 10-cut. Rejects from the secondary screens are diluted to 0.2% consistency and pumped to one line of tertiary screens. These are similar to the others except that the openings are 8-cut. Rejects from the tertiary screens go to jordan refiners, then are screened and used for coarse wrapping grades of paper. The normal daily screen capacity is 180 tons, but up to 250 tons have been handled.

In the meantime, all the stock that passes through the slots in the primary, secondary, and tertiary screens is thickened on four deckers. The decker is simply a wire mesh-covered cylinder rotating in a tile-lined vat filled with screened pulp. The cylinder is 48 inches in diameter, 120 inches long, and rotates at a peripheral speed of 120 feet per minute. A partial vacuum maintained within the cylinder causes water to flow through the mesh and fibers to be retained on the surface. This stock at a consistency of about 4.5% is removed by a rubber-covered self-driven roll, called a couch (*3*). A rubber and asbestos doctor removes the thickened stock from the couch.

The stock is then diluted by showers to about 3.5% and run into two screened stock storage chests made of tile-lined concrete (25 tons of air-dry pulp capacity). These are agitated as in the previous stock chests. From these chests the pulp can be pumped either to the kraft bleach plant or to the beater chests.

BLEACHING

All wood pulp, no matter how carefully made, has associated with it some lignin and certain color bodies of highly complex chemical nature. To make the brighter grades of paper, bleaching is necessary. This is done in two major steps: the lignin remaining from the digestion process is removed by treatment with chlorine and sodium hydroxide; and the brown color associated with kraft-cooked pulps is bleached by calcium hypochlorite. The latter is the most expensive of the bleaching chemicals. It is also least selective and most harmful to the pulp strength. Based on these facts, the Camas bleach plant is designed to produce a pulp fairly free of lignin before hypochlorite is used (Fig. 4).

Process flexibility is essential for coping with the various colors and wood species handled. A system of three-way valves and pipes along with the necessary chemical treatment equipment makes it possible to change the bleaching sequence in several ways. Any of the stages can be skipped, thereby using only that number needed for the particular grade and process. Pulp

Table II. Process Variables for Fully Bleached Kraft (80 G.E.)

	Cl_2, %/ton, air-dry pulp	*NaOH, %/ton air-dry pulp*	*pH at end*	*Temp., °F.*	*Consistency, % air dry*	*Retention time, min.*
4-Stage system						
1st chlorination	7.0	0	2	Cold	4.0	60
1st hypochlorite	4.5	1.9	7.5	90	13.3	30
1st caustic	0	2.1	8	160	11.0	60
2nd hypochlorite	1.2	0.3	7.5	90	5.5	210
8-Stage system						
1st chlorination	7.0	0	2	Cold	4.0	60
1st caustic	0	4.0	8	160	11.0	90
Soak	0	0	7.5	Cold	3.3	60
2nd chlorination	1.3	0	3	Cold	3.3	60
2nd caustic	0	1.3	8	140	11.0	90
1st hypochlorite	1.7	0.3	7.5	90	13.3	210
2nd hypochlorite	0.1	0	7.5	90	5.5	180
SO_2 flash	0	0	5.5	Cold	3.3	60

specifications in this connection are based on a standardized reflectivity or brightness as measured by a General Electric brightness tester (*14E*). The higher the reading on the 0 to 100 instrument scale, the brighter or more completely bleached is the pulp. This instrument uses a specially prepared magnesium oxide as a full-scale standard of 100. On this scale, "full bleached" pulp will fall between 80 and 90.

When semibleached pulp (30 G.E. or 65 to 70 G.E. brightness) is being made, more than half the plant is bypassed. For instance, 30 G.E. pulp requires only one-stage bleaching with high density hypochlorite. The higher grades of semibleached kraft are bleached in three stages: chlorination, caustic extraction, and hypochlorite treatment. Fully bleached kraft pulp (80 G.E.) is normally done at Camas in four stages (Fig. 4).

Additional bleaching stages have been built into the

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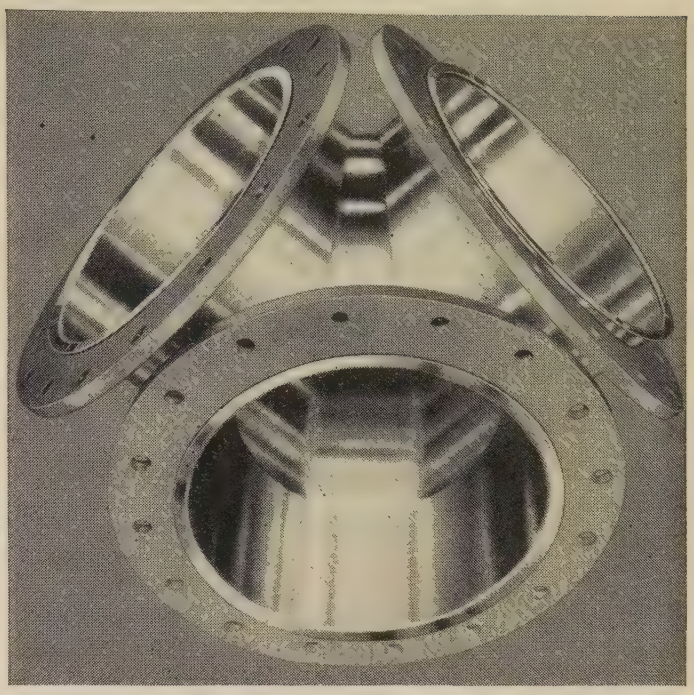

A fabricated Monel tee, used as a connection between a cycling chest, dump chest, and pump from the hydrapulper at the Maxwell Paper Company, Franklin, Ohio.

The tee measures 16 in. O.D. and is made of 14-gauge Monel. Fabricated by BROWN-SINGER CO., Middletown, Ohio.

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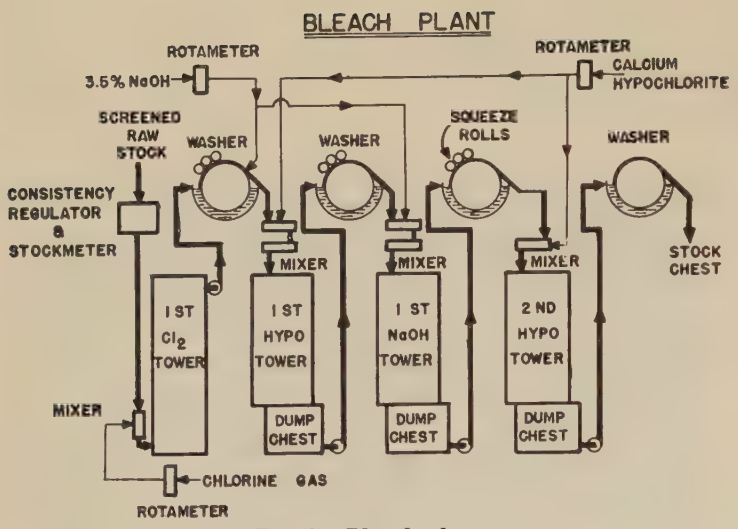


Fig. 4. Bleach plant

plant so that, if desired, an eight-stage fully bleached high-purity kraft can be made. These stages consist of a low-consistency chlorination; high-consistency caustic treatment; water soak; second low-consistency chlorination; second high-consistency caustic treatment; high-consistency hypochlorite treatment; low-consistency hypochlorite treatment; and for final whitening, treatment with sulphur dioxide. Rotary drum washers are provided between each of these stages to remove impurities before undergoing subsequent treatments.

As an example of common practice at the Camas plant, a four-stage process is described in detail (Fig. 4). Statistics are based on daily production of 150 tons of fully bleached 80 G.E. kraft pulp (Table II).

The pulp is pumped from the brown stock storage to a consistency controller (*24E*) and a recording stock meter (*26E*), which measures the production rate for the bleach plant. The stock is mixed (*27E*) with chlorine gas, then enters the bottom of a tile-lined concrete chlorinating tower where it is agitated by rubber-covered arms on a central top-driven vertical shaft.

The stock issuing from the top of the tower is pumped, washed (*25E*), and thickened to 17% consistency by squeeze rolls on the washer cylinder (Fig. 5). A 3.5% sodium hydroxide solution is added immediately after the last squeeze roll to neutralize the stock and prevent the hypochlorite from going acid in the subsequent stage.

The stock drops into a two-tier double shaft high density mixer (*28E*) where calcium hypochlorite solution (0.31 pound of chlorine per gallon) is added. The pulp color is broken down by oxidation in the first hypochlorite tower (tile-lined concrete). A rubber-covered scraper agitates only the bottom pulp. A variable speed screw discharge (*29E*) forces the pulp into a chest where water dilutes it to pumping consistency.

The pulp is washed, thickened, and discharged into the first caustic tower, which is identical to the first hypochlorite tower. A 3.5% sodium hydroxide solution is added at the mixer. After being retained for 60 minutes, the stock is diluted in the dump chest and pumped to the third washer where it is thickened and dropped into the second hypochlorite tower. This is similar to the first hypo tower, but its larger capacity permits a longer retention time. The stock is diluted in a dump chest and pumped over the final washer and transferred to the bleached kraft stock chest. From here it enters the beater room parallel to the unbleached stock.

Process variables for making 80 G.E. brightness stock in both four- and eight-stage systems are compared in Table II.

The operation of a bleach plant is continuous so it is apparent that any change in stock consistency or bleachability (*21*) must either be corrected at once or compensating chemical treatment started. Tests used for control are consistency, bleachability, pH, and residual chlorine. Flow rates of chlorine, sodium hydroxide, and calcium hypochlorite are measured by area-type meters (*9E*). Instruments record temperature, flow rate, and pressure.

BEATING AND REFINING

A major part of the transition between pulp and paper takes place in the beater (*19E*). Although it has been eliminated in some newsprint and kraft mills in favor of other types of refiners, the beater remains in most mills as the machine that "makes" the paper. Just how this comes about has been the subject of many theories since the first Hollander beater was invented about 1680 (*16*). Nobody really knows exactly what takes place during beating or why. The only way to tell what the beater or refiners are really doing is to see what effects they have on the character of the paper made from the stock. This is admittedly a circuitous way to control such an important function (*17*).

The present tendency is to consider beating as effecting physical rather than chemical changes in the pulp. Among these postulated are: fibrillation; decrease in thickness of cell wall due to removal of fibrils; transverse cutting (shortening); collapsing of fibers; longitudinal splitting of the cell wall, resulting in opening up fibers as ribbons; decrease in fiber stiffness; and decrease in rate of drainage of water through mats of fibers. Paper made from beaten fibers will be stronger, less opaque, or of more uniform formation; it will have greater density, because of greater shrinkage of wet web, and thus be less porous (*9*). The first six of these changes can be observed microscopically. Many re-

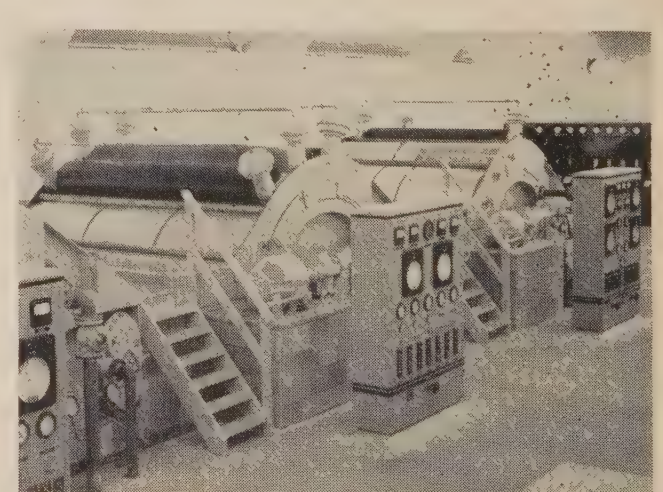
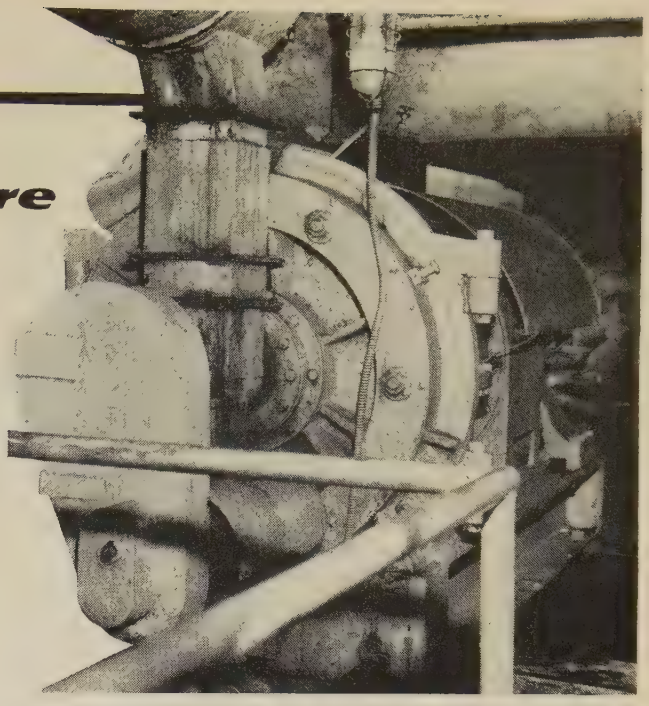
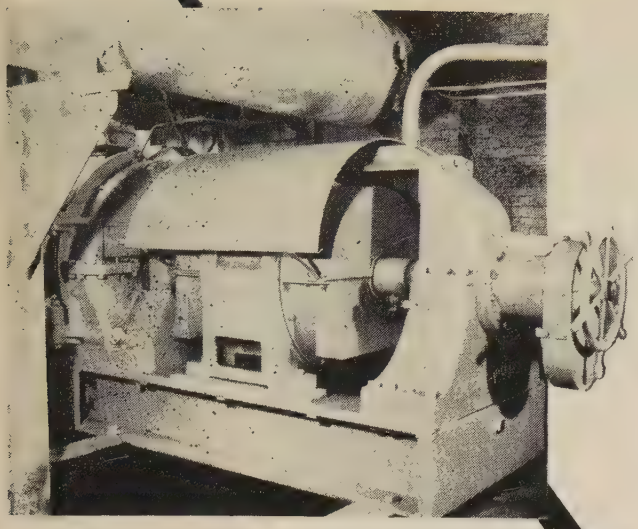


Fig. 5. Washers and squeeze rolls in bleach plant

Mills that compare

Refiners





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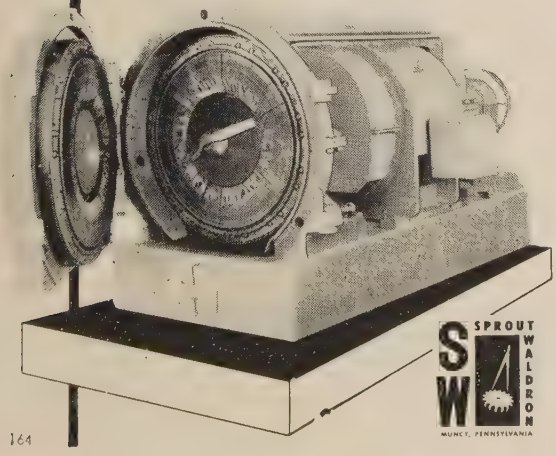

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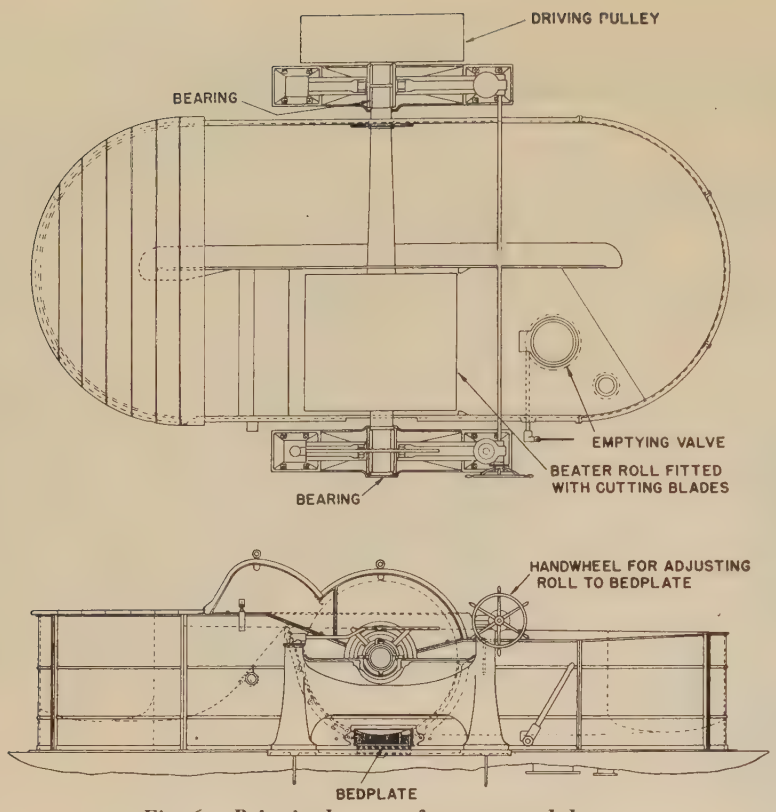
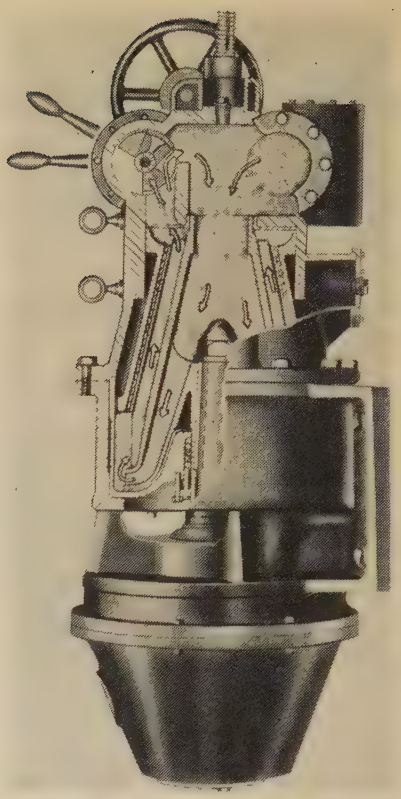


Fig. 6. Principal parts of paper-stock beater



Cross section of Morden Stockmakers

searchers claim that there is some interaction between water and the fibers. This so-called hydration can be interpreted in many ways. Pulp and paper vocabularies, textbooks, and technical papers are rife with ambiguous terminology. Perhaps semantic as well as scientific research is needed to clarify many problems of the industry.

When has a stock been beaten sufficiently to give optimum qualities to a particular grade of paper? The answer to this question is largely previous experience. There are tests of stock freeness nd aslowness which indicate rate of water drainage on the paper machine wire, but these do not tell the whole story. According to Sutermeister (*17*):

> There is no beater control test of general applicability, and the only way to tell the condition of the stock is to make it into sheets under carefully controlled conditions and test them for their physical characteristics. Even this is subject to so many influences that results in different plants are not strictly comparable. It would seem that in spite of all the study given to it, beating is still an art, and not a science.

Figure 6 shows the principal parts of a beater. A heavy hollow iron roll is fitted around its periphery with sharp steel fly bars. Wedge-shaped wooden segments are driven between the bars and when swollen with water hold the bars firmly in place. The bedplate is either stone or steel, the former is generally used for beating kraft. Most of the fibrillation takes place between the fly bars and bedplate. The roll is set on a spindle across the tub made of tile-lined concrete. The backfall is concrete covered with metal. A doctor prevents too much stock from being carried completely around the roll as this would reduce circulation and cut capacity. The stock circulates around a mid-feather in the center of the beater. The washer is an octagonal hollow screen covered with bronze cloth. During "washing" it is lowered one third of its diameter into the pulp; water flows through the screen, is scooped up by internal baffles, and discharged through a hollow shaft. This makes possible rapid increases in consistency.

Stock from the bleach plant, or unbleached stock directly from the decker chests following screening, arrives in the beater at 2.5 to 3% consistency. It is thickened to 5 or 6% in 10 minutes while the beater roll is up off the bedplate. Then the washer is raised and the roll lowered to the desired distance above the bedplate. Previous tests determine what surface speed of the roll will give maximum beating consistent with power consumption. Speeding the roll beyond this point accelerates the beating rate, but power demands are not proportional to the results obtained. Estimates indicate that above 60% of the power consumed is used just in circulating the stock (*4*).

The beating cycle for most kraft pulps is from 30 to 60 minutes. During this period, the beater also serves to mix any added chemicals. The two most commonly used are rosin size and papermaker's alum.

Rosin size is purchased in tank cars as a thick paste (80% solids) and prepared for beater addition by heating to 170°F., then running through an ejector where it is mixed with hot water and emulsified. The 8% solids emulsions if further diluted with water to 4%, at which concentration it is furnished to the beater. This size averages 25% free rosin acids with the remainder saponified.

Rosin size increases the resistance of paper to penetration by liquids such as water, ink, meat juices, and paste. Besides there being a number of theories on sizing (*18–20*), the very word "sizing" seems to mean all things to all men.

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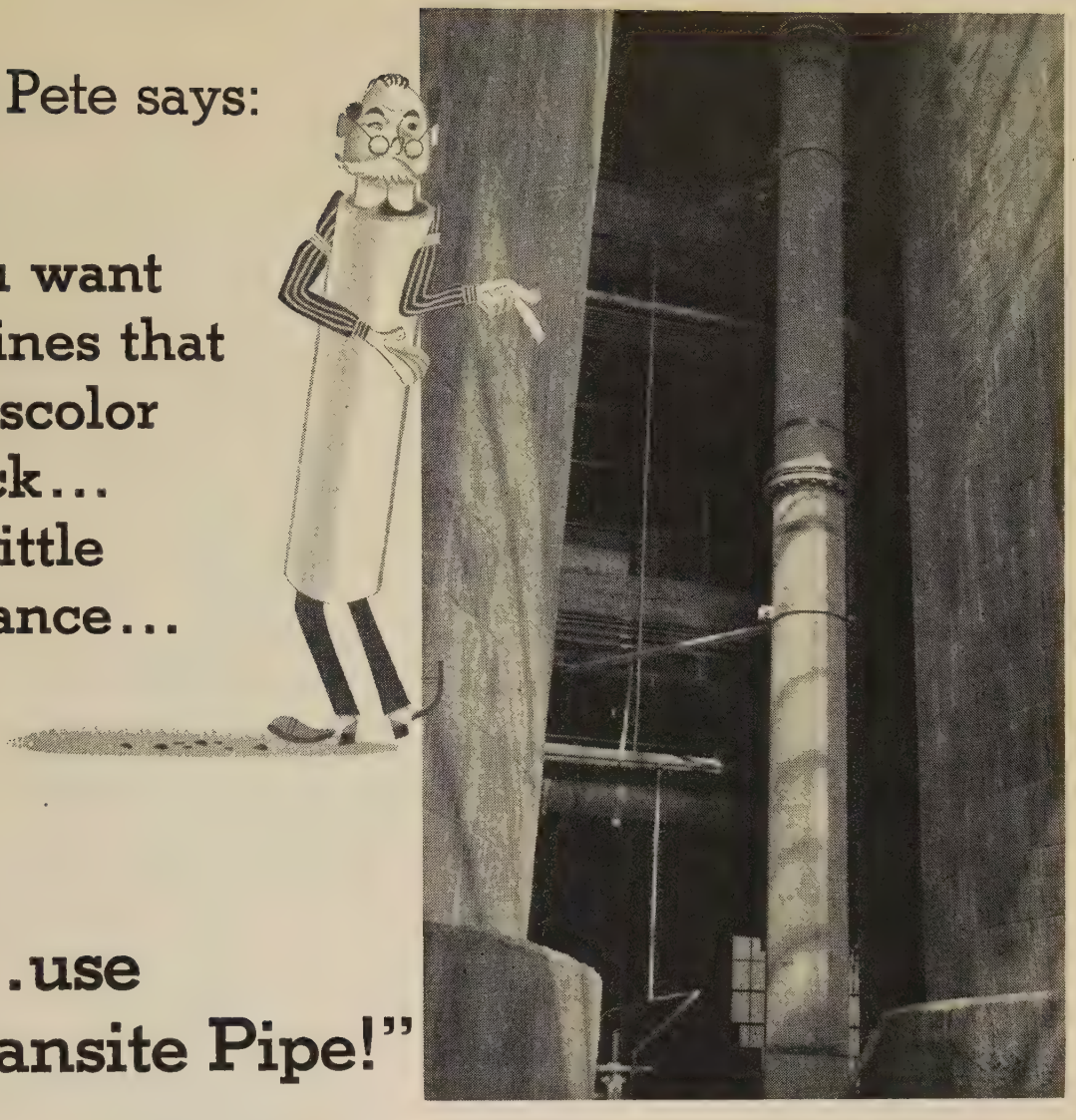

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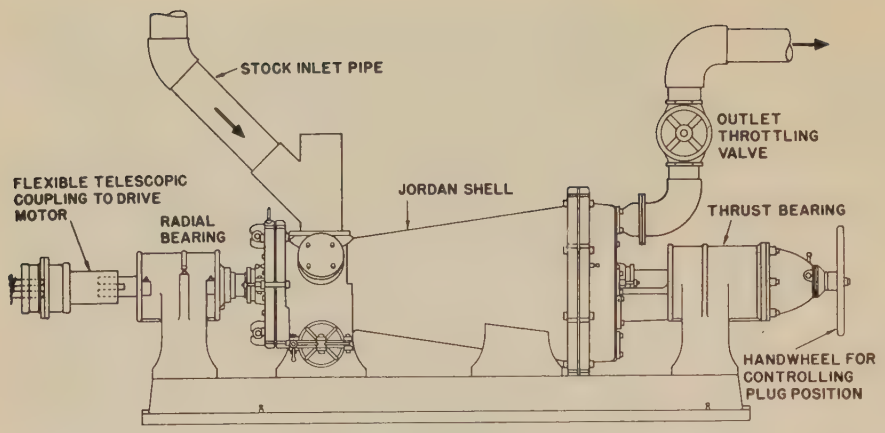

Fig. 7. Jordan refiner

It is not the purpose of this article to discuss in detail the theoretical aspects of any particular phase of papermaking, but rather to show the application of chemical engineering principles in a particular modern kraft mill.

Certain hard-sized sheets require more than the normal amount of sizing. A high free-rosin size (colloidal free-rosin acids protected by protein) is a more effective agent than ordinary rosin size. A wax-size emulsion may be used as a supplement for beater addition. The latter is bought as 50% solids and diluted with cold water to 0.5 pound per gallon (6%).

Loading pigments may be added to the beater stock. These include clay, talc, titanium dioxide, lithopone, and extended pigments (titanium dioxide coprecipitated with barium or calcium sulphate). Titanium dioxide and the extended pigments are necessary for brightening and opacifying waxing grades. These may also be used for brightening other grades, but less expensive materials such as clay, lithopone, and talc are preferred. For the cheaper writing papers, clay or talc opacifies and improves the writing surface. Addition of any pigment gives the paper more pliability and less rattle and resiliency.

Colors for paper are added in the beater after loading. Many types of aniline dyes (basic, direct, acid) may be used. These are added as concentrated solutions or in powdered form to be dispersed under the beater roll. The addition of any dye decreases the paper brightness, in spite of the apparent opposite effect when, for example, blue is added to white paper. Other colors used in minor quantities are the earth dyes (sap brown), certain chrome colors, and carbon black.

Papermaker's alum is $Al_2(SO_4)_3 \cdot 18H_2O$. It is added to the stock in the beater to precipitate the size and to set certain colors through adjustment of the pH. The retention and tint of many aniline dyes are affected by pH. For heavily loaded or pigments sheets, the colors are best retained at a pH below 5.5. For economic reasons and to avoid paper embrittlement, the pH is kept above 4 in the system.

If more alumina is required for setting the size without a concomitant lowering of the pH, sodium aluminate may be used. In the grades of paper made at Camas, it would be too expensive to use iron-free alum. Ordinary technical grade suffices. This is in contrast with mills making fine paper grades which would be darkened or otherwise adversely affected by traces of iron. For certain grades at Camas, a portion of the alum is added later at the stuff box, just ahead of the paper machine (Fig. 1).

At the end of the beating cycle, the roll is raised and the stock dumped through a trap door (just ahead of the roll) and into a rectangular tile-lined first primary beater chest (capacity 8 tons air-dry stock at 3.5% consistency). Internal construction with impeller agitation is similar to that of the receiving chests ahead of the screens.

The stock is pumped from the first primary beater chest through two refiners (*21E*) in series (Fig. 8) to supplement beating and then into a second primary beater chest. Now the stock leaves the beater room and travels 200 yards to the papermachine building where extra storage capacity is provided by a secondary beater chest identical with the others. The stock may be pumped through two more refiners (*21E*) in series and then to the machine chest, also identical with the previous three chests.

After passing through a consistency controller (*7E*), the stock is divided and passes through two to four jordan refiners (*18E*) depending on the grade being made. Figure 7 shows the internal construction. A central plug fitted with bars or knives turns inside a similarly fitted shell. Usually the knives in both shell and plug are slotted, keyed, or banded to the casting, and the space between them is filled with wood strips driven tight and swelled with water before the plug and shell knives are faced to true cones. Stock enters at the narrow end of the cone, passes between the whirling knives, and the centrifugal action forces it out the larger end.

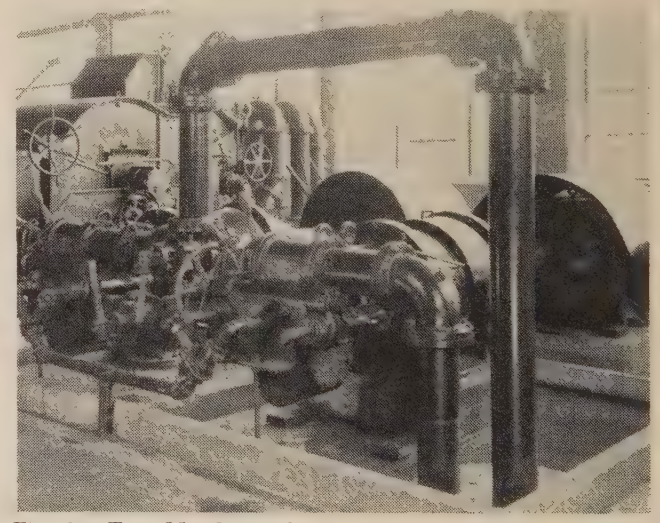

Fig. 8. Two Morden refiners in series; beater in background

Jordan refining can be changed in character and degree by adjusting the clearance between the plug and shell; changing the speed; changing the composition, width, and arrangement of the bars; changing stock consistency, hydrostatic head on the stock in the re-

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finer, and the temperature. Practice is moving toward higher speed and higher consistencies. Speeds range from 300 to 550 r.p.m., which give the large end of a 30-inch diameter plug peripheral speeds of 2300 to 4500 feet per minute. This is nearly twice that of beater rolls (*10*). Stock or stuff consistency to jordans is usually 3 to 3.5%. Looking toward higher capacity, with stronger jordan designs and better application of

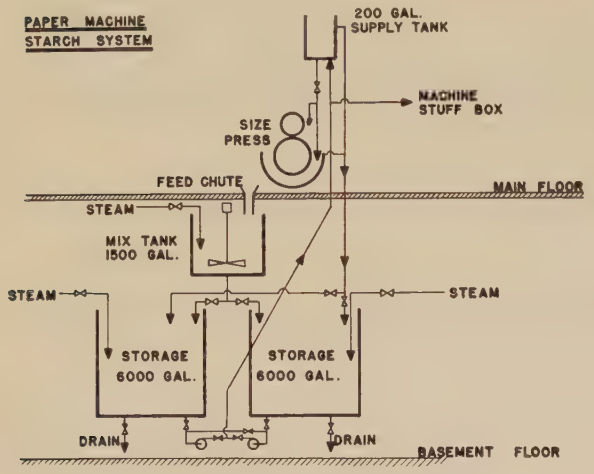

Fig. 9. Starch system

power, consistencies tend to move upward. In the interest of sheet formation, with improved bearings, dynamic balance, and stronger materials, speeds also tend to move upward, since high speeds affect formation with less slowing of stock then slow speeds.

Jordans are primarily designed for cutting action on the fibers and should not be overloaded by doing the work of hydration best accomplished in the beater. Bronze knives or bars remain sharp on the leading edge and have more of a cutting action on the fibers, whereas steel bars tend to round over on the edges producing a brushing action. Monel metal bars have an intermediate effect (*4*).

At Camas, the jordans accomplish the final cutting and refining prior to the paper-machine. Operation of the jordans is under the jurisdiction of the paper machine tenders who use this means to develop and control the final strength of the pulp.

The stock is pumped to the stuff box where the basis weight of the final paper is controlled. The stuff box has three compartments. The stock from the jordans enters the center one. An adjustable gate at the exit end allows just enough flow for the particular sheet of paper being made. Excess stock from the jordans overflows into the third compartment and is returned to the machine chest (Fig. 1).

Many materials that are important in establishing the final paper properties may be added at the machine stuff box. Certain characteristics of machine operation—for example, drainage through the wire and pH of the final sheet—require close control of the pH. Experience shows that the instrument-controlled (*6E, 22E*) addition of alum at the closest possible point to the paper machine provides the very narrow range of pH necessary for stock going to the machine.

Cooked starch solutions may be added at the machine stuff box to increase the dry bursting strength of the paper. Figure 9 shows the flow sheet and equipment used to add the starch solutions either at the machine stuff box or later on the paper-machine size press. Temperature and liquid level are recorded in the mix tank. In each of the storage tanks the temperature is recorded and controlled and the liquid level is indicated.

Melamine-formaldehyde resins may also be added at the stuff box if high wet-strength characteristics are desired in the finished sheet (*1, 7, 8*). Equipment used is similar (*2*) to that of the starch system with the following differences: A small amount of hydrochloric acid is added to the mix tank; the dilute resin colloidal suspension is stored in four 3000-gallon tanks without steam lines; only one transfer pump is used between storage and the 200-gallon supply tank; the resin is applied to the stock in the machine stuff box only, not at the size press.

From the stuff box, the stock usually goes directly to the fan pump (*40E*) where the consistency is reduced from 3% to 0.3 to 0.5% by addition of white water. This is the water that drains through the mat of fibers on the paper-machine wire. It is called "white water" because of its milky appearance which is due to suspended fine fibers and other solids. Because of the extremely high dilution of the stock at this point, the fan pump (*40E*) must be of high capacity—250-hp., 500-r.p.m., 13,000-gallons-per-minute, 40-foot head.

If metallic and other foreign particles are objectionable in the finished sheet, the stock may be passed from the stuff box and through an auxiliary mixing pump (*41E*)—125-hp., 700-r.p.m., 10,000-gallons-per-minute, 40-foot head. Here the stock is diluted with machine white water and then passes through a series of Dirtecs (*3E*). Foreign particles heavier than normal stock fibers are separated by centrifugal action as the stock spirals up long narrow cylinders. Only enough units are used to give optimum separation or removal of dirt. Stock from the Dirtecs passes to the main fan pump where additional white water brings the consistency down to 0.3 to 0.5%. A series of supplementary screens is available (*4E*).

THE FOURDRINIER PAPER MACHINE

The diluted stock enters the paper machine headbox (*1E*) by means of distribution pipes entering each end. The headbox quiets the flow and ensures uniform distribution of fibers by means of a series of internal baffles and rectifier rolls, which are perforated, rubber-covered, and rotate at 20 to 30 r.p.m. in the stock.

The stock flows from the headbox, across a rubber covered cloth apron, through an adjustable rectangular orifice—the slice shown in title photo—and onto the fourdrinier paper machine wire directly over the breast roll. Figure 10 shows details of the No. 15 fourdrinier at the Camas mill. All subsequent data refer to this specific machine (*2E*).

The fourdrinier "wire" is a fine mesh bronze wire continuous cloth, 60 by 44 mesh weave, 152 inches wide and 105 feet long (periphery), which passes over the breast roll to pick up stock flowing through the slice. The linear speed of stock flow should be about the same as that of the wire. If the stock moves too fast, rolling of the fibers takes place to form a square or nondirectional sheet. This has poor appearance and may be undesir-

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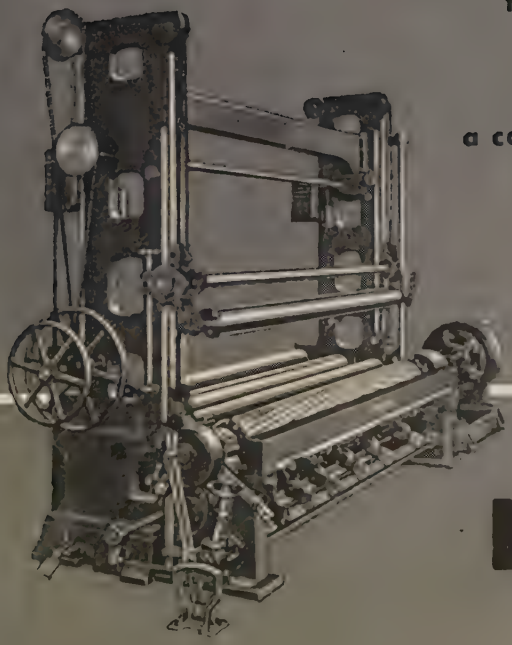

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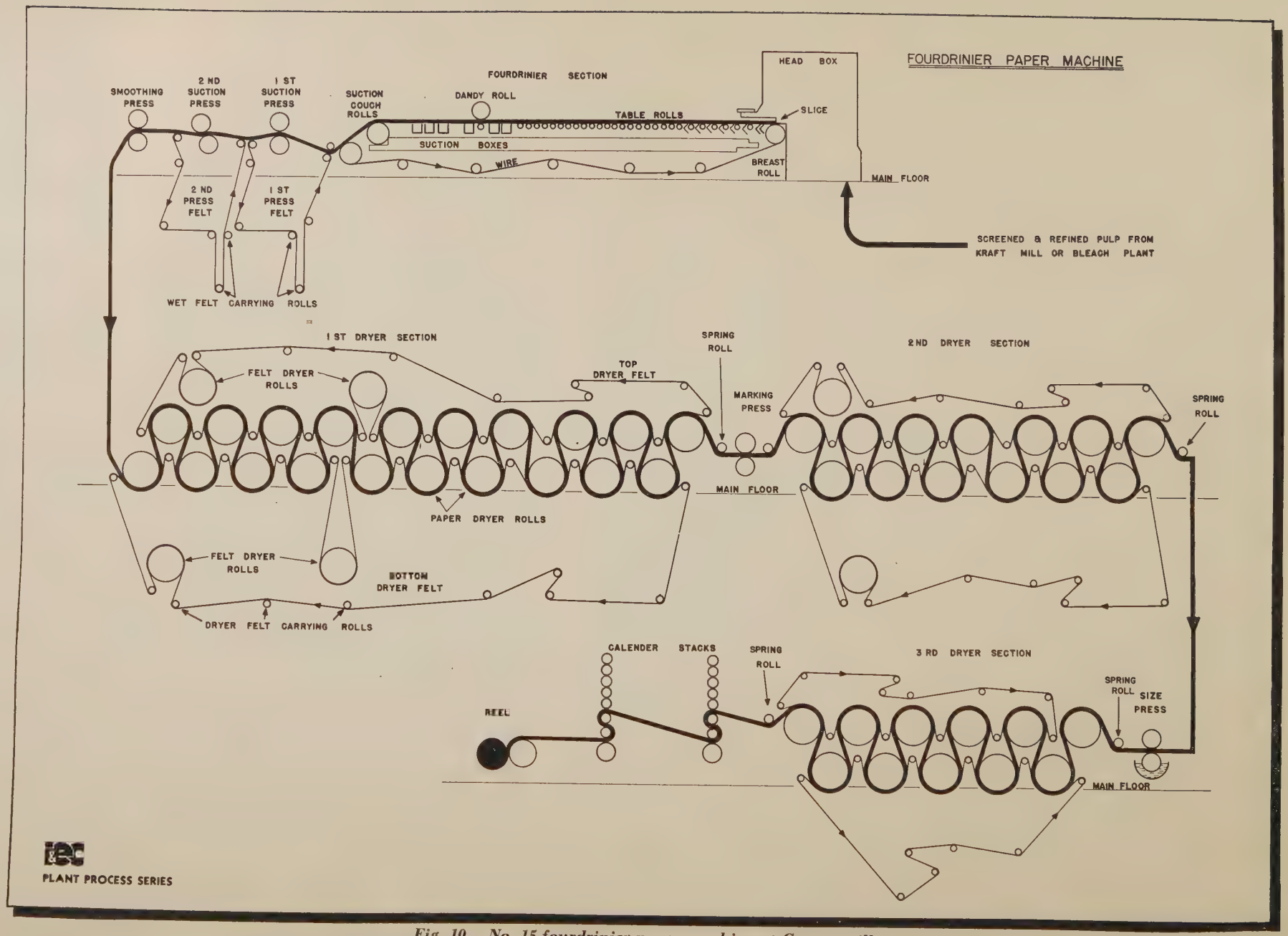

Fig. 10. No. 15 fourdrinier paper machine at Camas mill

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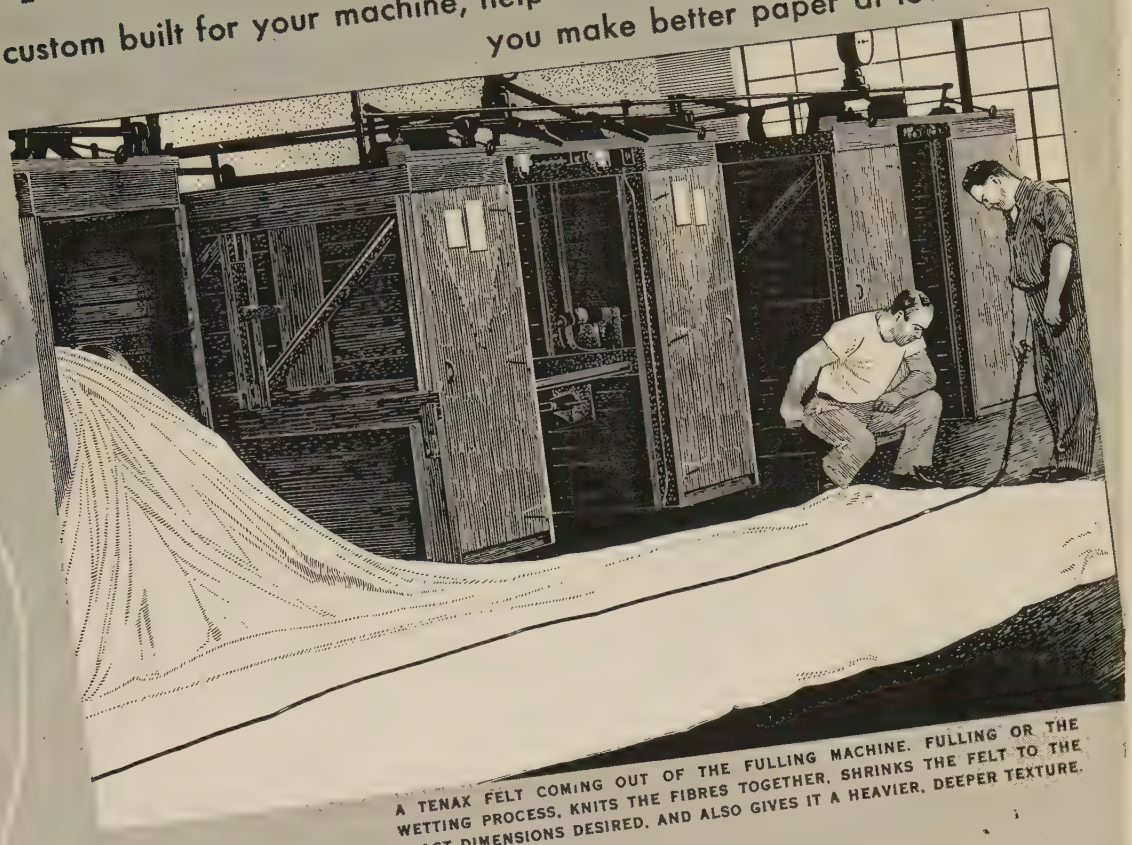

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able in certain converting operations, such as cutting. If the stock flow to the wire is too slow, there will be drag of the fibers producing a highly unidirectional sheet that splits easily and would not be good for paper bags, for example.

Stock flow onto the wire is controlled by the opening of the slice and the stock level in the headbox. To speed up the flow, either the slice can be narrowed or the stock volume increased in the headbox by adding more white water at the fan pump. This lowers the consistency since the amount of pulp coming from the stuff box is uniform. Adjustment of the slice is very delicate, and the general sheet characteristics (tensile, tear, burst, formation, and basis weight) are very sensitive to small changes in slice settings. Unequal distribution of stock coming through the slice can be corrected by varying short increments of the 146-inch flexible stainless steel bar that comprises the slice.

About half the length of the machine wire is supported on a series of thirty-two table rolls followed by seven suction boxes. This constitutes the section on which the wet web or sheet is formed. (Data describing all the different fourdrinier rolls are included in Table III.)

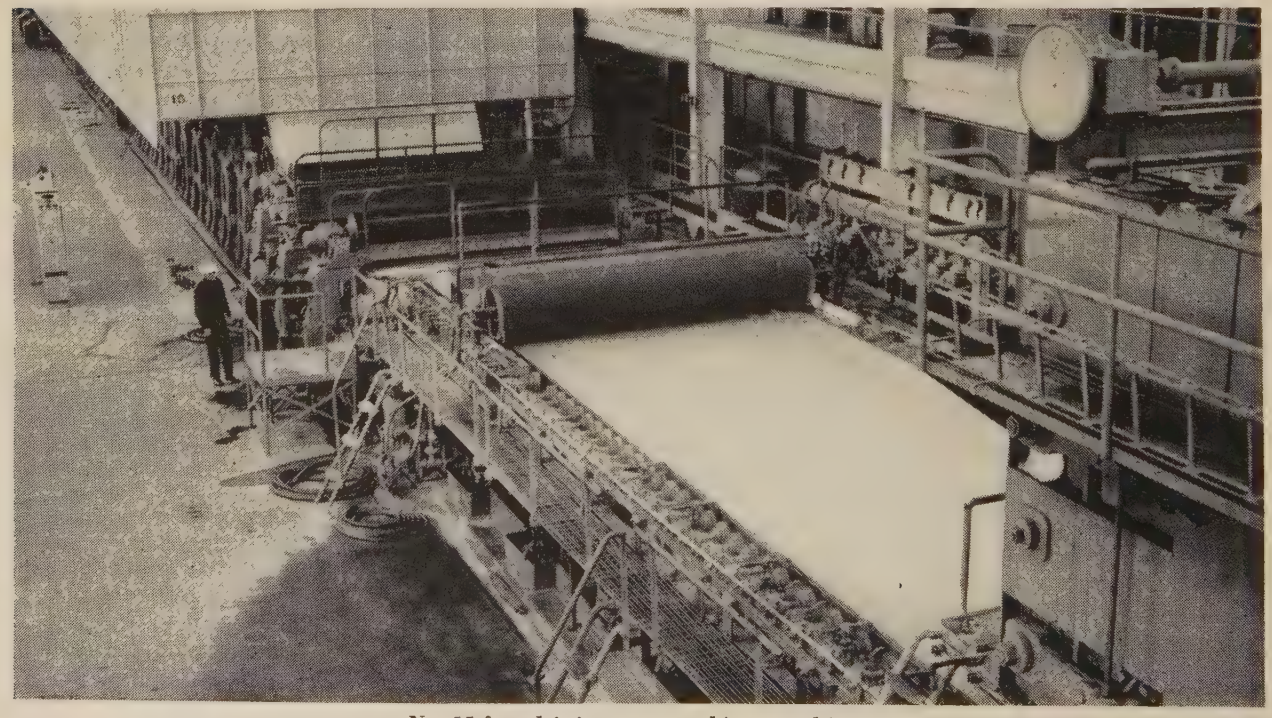
As soon as the stock flows onto the wire, white water drains through by gravity, leaving a wet mat on the wire surface. Because of surface tension, the table rolls also aid in drawing water from the sheet. Resting lightly on the wire near each edge is a narrow strip of stainless steel with rubber lip. These "deckle" strips extend for about half the length of the table roll section and prevent the pulp from flowing off the edge of the wire during the initial stages of sheet formation. Sidewise oscillation of the breast roll and table roll section shakes the wire perpendicular to its direction of travel. This improves the fiber distribution or formation of the sheet. Metal strips called deflectors are placed between each of the first six table rolls to divert the heavy flow of white water into the wire pit.

When the sheet reaches the suction or flat-box section, most of the free water has been removed. A vacuum (6 to 8 inches of mercury) pulled on these six stainless steel maple-faced boxes draws more water (about 25% of the total) out of the sheet and through $^5/_8$-inch round holes (rows 0.75 inch apart set on 1-inch centers). The suction boxes move eccentrically at 2.5 cycles per minute to eliminate any local vacuum irregularities that would affect the sheet formation and also to avoid wire drag.

A dandy roll, consisting of a light cylindrical frame covered with a bronze wire of mesh similar to the machine wire, runs free on the top of the sheet after the second suction box and is supported by a table roll. It improves the formation, smooths the sheet, reduces the fuzz, and prevents sticking on the first press roll. Immediately following the suction boxes, the wire travels over two perforated suction couch rolls (18 inches of mercury vacuum) which remove more water from the sheet. These rolls, known as the primary and main couches, drive the wire and therefore do not exert the frictional drag typical of the suction boxes. The sheet is removed from the wire at the main couch and transferred to the first press wet felt (wool blanket) which transports it through the subsequent press section.

After the wire leaves the main couch, it passes over a series of supporting return wire rolls back to the breast roll under the slice (Fig. 10). One of the supporting rolls is mounted on a movable bracket to permit adjustment of wire tension. All rolls must be furnished with showers and doctors to prevent wire damage caused by stock collecting on the roll surfaces.

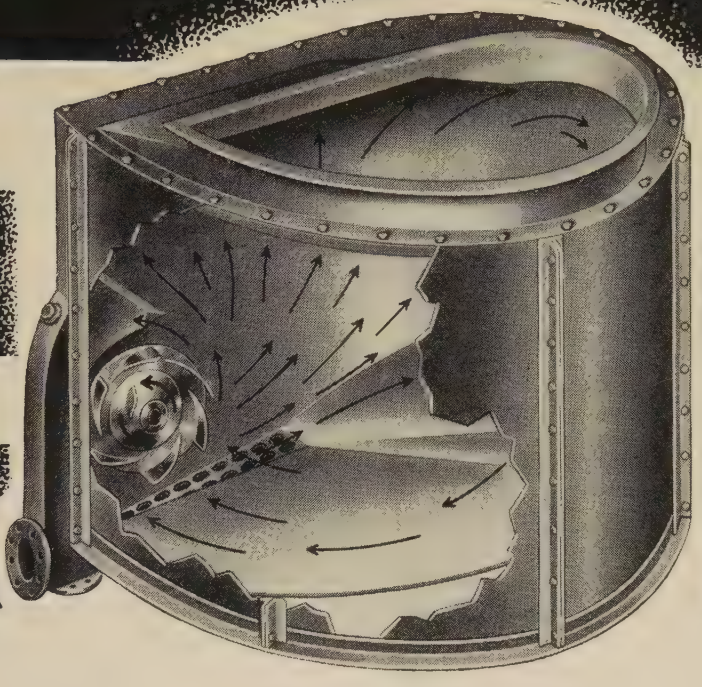
After leaving the wire, the wet sheet is carried on wet press felts through two sets of suction press rolls to remove more water. Effort is made to remove as much water as possible in the press section since evaporation of water by steam in the dryers is more expensive. The limiting factor in mechanical water removal by presses is the point where loading of the rolls "crushes" the sheet.



No. 15 fourdrinier papermaking machine

Cowles PULPER

Outstandingly Faster, Finer at ALL Levels



Outstandingly fast on every kind of furnish

Minimum Hp/T/D where ultimate defibering is required

Good submergence under all conditions

Effective operation at all levels —no exceptions

The Cowles Side-Drive Pulper is new in name only. It is a machine with years of practical mill-test behind it . . . constantly refined to produce the ideal pulper.

The aim has been to develop in high degree the advantages that all mill operators want, namely: speed, thoroughness, low power consumption; good operation and submergence at all levels; good circulation under all conditions; no dead spots, no clogging or jamming with any kind of furnish; fast, clean and complete dumping; and simple, rugged construction. Above all, minimum building and floor space requirements: *the Cowles pulper requires less head room than any other pulper now on the market.*

Side-drive design permits circulation of high consistencies with small high-speed impellers, sharply reducing the cost of the impeller and drive mechanism. A steeply-sloped bottom promotes rapid drainage; will handle batches from ⅓ to full size with equal efficiency. There is virtually no splash at any operating level; the submergence is exceptional at all levels and consistencies.

Made in a variety of sizes and designs to suit all conditions.

Write or wire for details of rugged construction and proved hydraulic design.

The Cowles Company
INCORPORATED
1012 Trackside Cayuga, N. Y.

Associates: Shartle Bros. Machine Co., Division of Black Clawson Co., Middletown, Ohio • Alexander Fleck, Ltd., Ottawa, Ont.

The sheet on the wet felt is passed between the nips of the first press rolls. Both are rubber-covered and the bottom one is perforated for suction. The sheet is transferred via a second press wet felt to two press rolls similar to the first pair.

At the same time, the two wet felts travel over a series of wet felt carrying rolls, one of which is provided with a movable bracket to adjust the tension. Suction boxes condition each felt by removing water, bringing out the surface nap, and cleaning the surface of wax and size.

From the second suction press, the sheet passes through a smoothing press without felt support or cushion which gives a certain amount of finish to the surface. The sheet (at about 65 to 70% moisture content) is now ready for the 1st drier section.

Something should be said here about roll design. In order to keep the sheet of paper uniform, it is necessary to "crown" a single roll, or the lower roll of a pair, by making its diameter in the middle just enough larger than at the ends to make up for the sag in the middle caused by the weight carried by the roll in the machine (*11*). Table III indicates the crowned rolls on the fourdrinier.

It is important here as elsewhere on the machine to have the draw carefully adjusted. "Draw" is the tension (stretch) on the paper as it passes from one section of the machine to another. There must be a certain tension, or draw, between couch rolls and the first press rolls, between the press sections, the last press and driers, between driers and calender, and between calender and reels. The term is especially applicable to the wet end of the fourdrinier where the paper gets most of its longitudinal stretching, which is caused by each press running more rapidly than the preceding one (*12*). Actual tests have shown that comparatively small changes in draw have produced a 30% change in tensile strength of the paper (*5*).

The paper is dried on a series of drier rolls arranged in three sections of 20, 13, and 11 rolls, respectively. The sheet of paper passes around each roll in succession, being held in close contact by a heavy drier felt or canvas (0.25 inch thick, cotton) which is sufficiently porous to allow ready escape of water vapor. The felt in the first section is dried by four separate drier rolls; there are two in the second section. These are identical to those used to dry the paper sheet (Table III).

A pair of rolls, usually functioning as a secondary smoothing press but termed a marking press at Camas, is located between the first and second drier sections. Small spring rolls maintain constant draw on the paper. A size press is positioned between the second and third dryer sections. The two rolls are equipped with a pan and shower arrangement for the application of suitable sizing materials to the sheet surface. The marking or secondary smoothing press and the size press are used to provide additional finish or caliper control (sheet thickness).

After leaving the last drier section, the sheet of paper is essentially dry, averaging 5 to 6% moisture. Finish is applied to the paper by passing it through a series of chilled iron calender rolls. Only the bottom roll is driven so that there is some slippage between adjacent rolls, producing an "ironing" action on the sheet. Some of the rolls are drilled for water or steam. The sheet is passed back and forth starting at the top of the stack. The desired paper finish is obtained by varying the number of roll nips; by auxiliary pneumatic loading; and by circulating water or steam through the calender rolls.

After passing through the second calender stack, the sheet is wound on a steel reel spool. When the roll attains the desired diameter, the paper coming off the machine is transferred to an empty reel spool. The full one is removed to the machine winder by means of an electric hoist. The paper is rewound usually on paper cores and at the same time it is slit to the desired roll length, the deckle edge is trimmed, defects removed, and splices made. The paper is ready for shipment in rolls or it is sheeted according to the specific demands of the customer. At Camas much of the paper is converted to bags, wraps, and other consumer shapes in another part of the mill. Paper is wrapped, stenciled, and made ready for delivery to railroad boxcars.

WHITE WATER AND BROKE RECOVERY

Since a large percentage of the fiber run onto the machine wire passes through and would be lost, the effluent must be conserved.

The white water passing through the wire at the table rolls and flat-box section carries about 10 pounds of fine fibers per 1000 gallons. This dilute suspension is collected in the wire pit directly beneath the fourdrinier. About 90% of the white water is returned to the fan pump to dilute the fresh stock entering the head box. The remainder passes into a tile-lined white-water chest (5000 cubic feet) from which it is pumped to a disk filter (*23E*).

About 90% of the fines are removed and returned as wet filter cake to the machine chest. A portion of the raw stock from this chest is mixed with white water just ahead of the filter. This is known as "sweetener stock" and provides the heavier mat on the filter wires for more efficient recovery of fines. As much filtrate as needed is pumped to be used on the paper machine as dilution water in the stock regulator, head box showers, or showers on the rolls in the fourdrinier section. Filtrate not used for these purposes overflows a weir to the sewer.

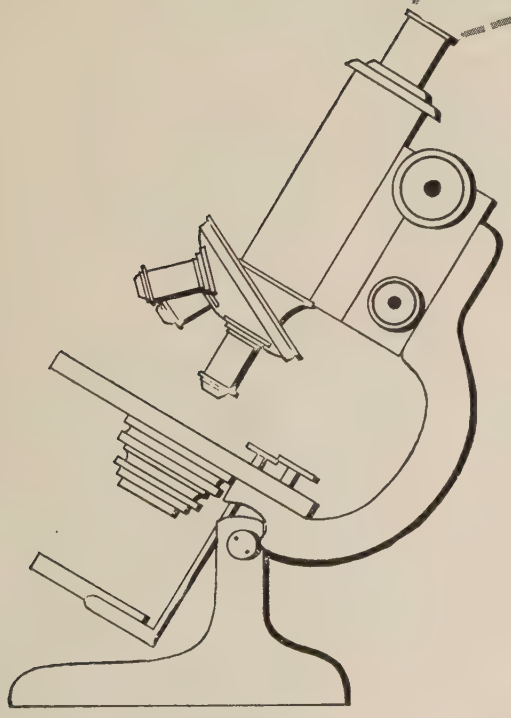
Facilities must be provided for handling "broke" or paper that has been discarded anywhere in the process of manufacture. This may accumulate during breaks on the paper machines; as trim or defective paper removed at the winder; or as off-quality paper made during changes. In the basement approximately underneath the machine winder is a broke beater which repulps all dry broke accumulated. After being thoroughly pulped in the broke beater, the stock is returned to the secondary beater chest.

If paper breaks on the machine, the sheet either drops off the last couch roll into the wire pit or is doctored off the first press roll while the tail consisting of a narrow width of sheet is taken through the drier section. The sheet coming off the wire is mixed with the stock entering the white-water chest. The sheet doctored off the first press roll is known as wet broke and is repulped in the broke beater.

During changes of paper grade on the machine, surplus stock drops into the white-water chest, then it is pumped to a tile-lined reclaim chest (1400 cubic feet).

kill these pests...

Typical slime-forming organisms



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Quality conscious paper mill superintendents know from experience that chlorination, engineered to their stock or white water requirements by Wallace & Tiernan, effectively eliminates such slime-induced troubles as pin holes, slime spots, and poor sheet formation.

Such results—which have saved up to 400 dollars per day in many mills—are possible because chlorination kills the organisms that *cause* slime. It may be used alone on the fresh water supply, or together with chloramines or other corrective chemicals on the white water, for effective slime control in such key spots as the showers, screens, deckers, and at many other points.

Wallace & Tiernan Chlorinators are particularly effective in slime control work because they are especially designed from over 35 years' experience to give low cost, efficient performance.

For a survey on the chlorination needs of your mill without obligation, call your nearest W & T Representative now and start your plant on the road to slime free operation.

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CHLORINE AND CHEMICAL CONTROL EQUIPMENT

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I-36

Stock from the broke beater, white water which cannot be used back in the system, and stock from the filter vat can be sent to this chest. From here the stock is pumped via float control valve to the mill's central reclaim system where it is used to make lower grade papers.

To ensure cleanliness of product, as well as maximum equipment life, great care was taken to select materials of construction which would resist corrosion and have no tendency to stain or discolor the stock. As mentioned before, all chests are tile-lined. Stock and water lines are either of wood or transite. Machine and equipment parts are of stainless steel or other special alloy, or micarta, or are rubber-covered.

MOISTURE CONTROL

To ensure proper moisture control of the sheet on the paper machines, the paper and felt drier rolls are divided into five sections. The first eight driers of the first section comprise the first control section. The remainder of the first section, together with the second and third drier sections comprise the second, third, and fourth drier control sections, respectively. The six felt drier rolls are controlled independently to form the fifth control section.

Moisture-laden air must be removed immediately from the vicinity of the sheet within the driers. This is done by a series of five 2-speed exhaust fans (*33E*) which draw the air from around the driers and blow it out the stacks. Moisture-laden air is prevented from collecting in pockets within the drier sections along the length of the machine. The air is provided by a separate fan and is heated by exchange with steam (*34E*).

Four heating and ventilating units provide a continuous supply of warm air in the upper part of the machine room. This, together with the air provided through the nozzles, provides fresh air to the drier section.

In addition to the moisture control afforded in the four drier sections, the final sheet moisture and finish can be regulated, if necessary, by sweating the final drier and spring rolls. Sweat is caused by passing cold water through these rolls. Water may be added directly to the surface of the sheet on the first calender stack by means of a water box for finish or caliper control. Each calender stack roll is fitted with a hollow center which can pass either water or steam. Under normal operation, if used at all, water is run through the first stack rolls and steam in the second. The production from the paper machine is largely dependent on the mount of drying possible.

INSTRUMENTATION

The philosophy which guides the instrumentation on the No. 15 fourdrinier machine is simple and practical. In the first place, the operator must know what the instrument does, how it operates, and how it affects his product. In the second place, for maximum effectiveness the instruments and control levers must be easily accessible; should be in sight of the control point; and should be in a natural location regarding the process.

It is necessary to control the pH at the stuff box in order to set the sizing agents and to control drainage of water through the stock on the wire. Alum is added at the stuff box and the pH is measured (*22E*) at the exit side of the fan pump. This represents a time lag of 5 to 7 seconds. It is necessary to have proportionate control and reset (*6E*, *10E*). Instead of open and shut valve control on the alum line, the valve opening is directly proportional to the pH reading above a set value ranging from 4.5 to 6.0, depending on the stock.

At the running speed of the No. 15 fourdrinier, the level of stock in the headbox could not be read accurately with a sight glass. A bubble-type system dispenses with the sight glass and also records any level changes.

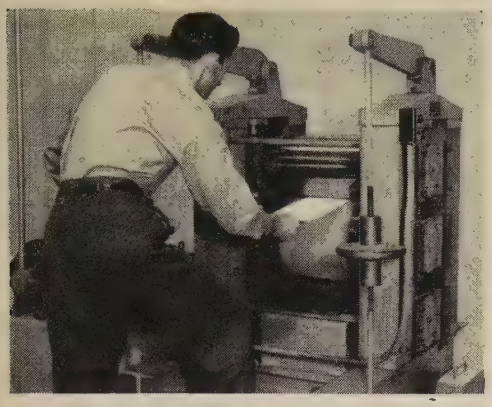
The vacuum in the suction boxes must be controlled accurately because of the large suction area and, more important, because of the danger of wire breakage during a slowdown. In this case, pulp would pile up over the suction boxes; wire wear would be excessive; with the end result that the stock might be pulled into the boxes with wire rupture (freezing of the wire). A straight proportional controller adjusts the vacuum by operating a bypass valve around the vacuum pump.

Table III. Rolls on Fourdrinier Papermaking Machine

Location on Machine	*Material of construction*	*Diameter, in.*	*Face, in.*	*Crown, in.*
Rectifier rolls in headbox (2)	Rubber covered	$12^{11}/_{16}$	$145^{1}/_{2}$	...
Breast roll	Brass covered	25	156	...
Table rolls (32)	Micarta	8	156	...
Dandy roll	30-Mesh bronze wire covered	24	156	...
Suction couch rolls (2)	Cast perforated bronze case	30	156	...
Return wire rolls (5)	Brass covered	$12^{15}/_{16}$	$155^{5}/_{8}$	...
Wet felt carrying rolls (14)	Rubber covered	$11^{1}/_{4}$	164	...
1st Press top roll	Rubber covered (plastometer 45) (*31E*)	28	157	0.070
2nd Press top roll	Stonite	28	$157^{3}/_{4}$	0.015
1st and 2nd Press bottom suction rolls (2)	Rubber covered	28	$163^{1}/_{2}$	0.040
Smoothing press top roll	Rubber covered (plastometer 21)	28	157	0.013
Smoothing press bottom roll	Stonite	27	$157^{1}/_{2}$	0.015
1st section paper and felt drier rolls (24)	Cast iron	60	$154^{3}/_{4}$	...
1st section dryer felt carrying rolls (39)	Steel	$11^{1}/_{4}$	$154^{3}/_{4}$	...
Spring rolls (5)	Brass	$12^{27}/_{32}$	$155^{1}/_{2}$	...
Marking press top roll	Rubber covered (plastometer 8–10)	28	157	0.012
Marking press bottom roll	Steel	28	149	0.012
2nd section paper and felt drier rolls (15)	Cast iron	60	$154^{3}/_{4}$	...
2nd section drier felt carrying rolls (30)	Steel	$11^{1}/_{4}$	$154^{3}/_{4}$	...
Size press top roll	Rubber covered (plastometer 24–25)	27	157	0.024
Size press bottom roll	Rubber covered (plastometer 14–15)	28	157	0.011
3rd section paper drier rolls (11)	Cast iron	60	$154^{3}/_{4}$	...
3rd section dryer felt carrying rolls (23)	Steel	$11^{1}/_{4}$	$154^{3}/_{4}$	...
Calender bottom rolls (2)	Cast iron	28	149	0.015
Calender rolls other than bottom (14)	Cast iron	15	144	...
Reel drum	Cast iron	36	144	...

KAOLIN CLAY FACTS... *for Paper Makers*

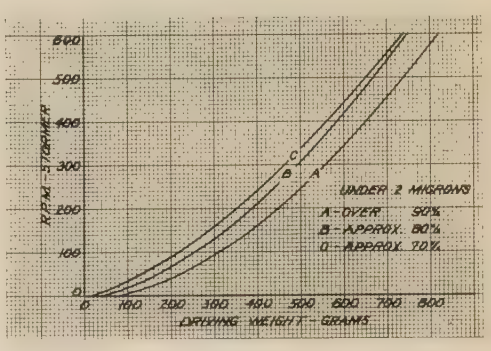
NUMBER 5 OF A SERIES FROM THE J. M. HUBER CORPORATION LABORATORIES



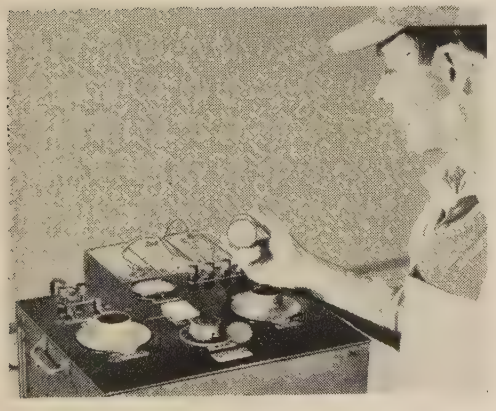
Calendering coated sheets in an Appleton Super Calender.

WHAT AFFECTS THE FLOW OF A COATING MIXTURE? The flow characteristics of coating mixtures during preparation and subsequent application to the surface of the paper largely depends upon the so-called "working properties" of the clay. In many modern coating operations, such properties are considered just as important as clay brightness and finishing characteristics. Mineralogical composition, concentration of clay, particle size, particle shape and the inherent ability of the clay to produce fluid suspensions through the mechanism of base exchange, all influence the flow characteristics of clay-water systems. The selection of coating clays on the basis of flow characteristics depends on the type of coating operation. Those processes requiring highly concentrated clay systems use the so-called "free flowing" clays, while the more viscous type of clays are often preferred in coating operations where high concentrations are not essential.

PARTICLE SIZE INFLUENCES CLAY-ADHESIVE FLOW In clay-adhesive mixtures, particle size is the predominant factor in controlling flow characteristics. The adjoining figure, compiled from data gathered by Huber technicians, shows the flow properties of coating colors prepared with a commercial grade clay from a selected crude. The formula used was 57.5% total solids with 17 parts of commercially modified starch to 100 parts of clay. In both starch and casein colors, the finer clays produce the more viscous mixture.



Effect of Clay Fineness on Flow Characteristics of Crude Clay No. 4 in a Starch Color (57.5% Total Solids).



Determining clay brightness with the General Electric Reflection Meter.

HOW COMMINUTION AFFECTS CLAY BRIGHTNESS Extensive studies in Huber Laboratories have shown that the degree of comminution affects the brightness of a clay. Coarsely ground clay has the lowest rating of all, while clays which are progressively refined through finer and finer screens show better and better brightness ratings on the GE Reflection Meter. A detailed study of this subject was made in the Huber Laboratories and published under the title "Brightness Determination of Paper Clays," by N. Millman, in the Paper Trade Journal, September 23, 1948.

Degree of Comminution	% Brightness
Lump clay crushed in a mortar	81.5
Lump clay passed through 60 mesh screen	82.1
Lump clay passed through 100 mesh screen	83.3
Lump clay passed through 150 mesh screen	84.1
Lump clay passed through 200 mesh screen	84.2

Have you received your copy of "Kaolin Clays and Their Industrial Uses"—new Huber 144-page technical reference book? Write on your company letterhead.

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J. M. HUBER CORPORATION, *342 Madison Avenue, New York 17, N. Y.*

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ONE OF THE WORLD'S LARGEST CLAY PRODUCERS

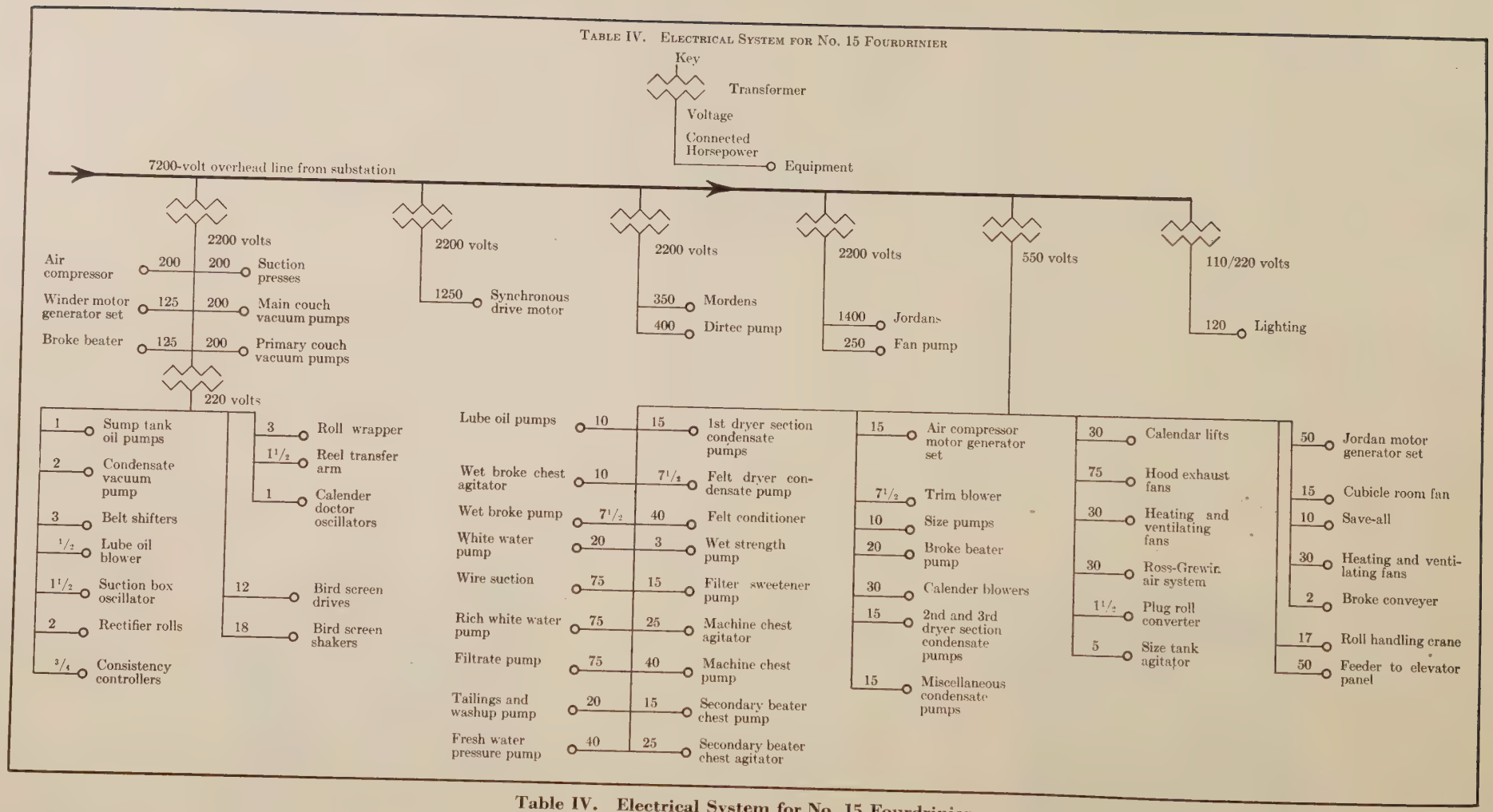
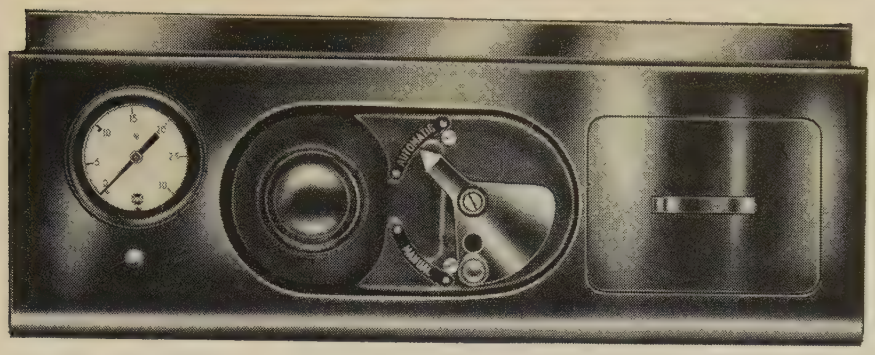
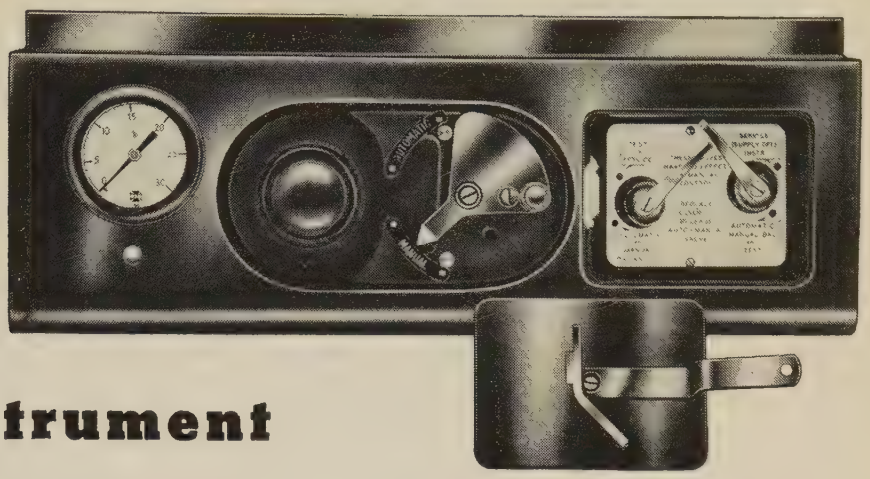


Table IV. Electrical System for No. 15 Fourdrinier
Key
Transformer
Voltage
Connected Horsepower
Equipment
7200-volt overhead line from substation
2200 volts
Air compressor 200 200 Suction presses
Winder motor generator set 125 200 Main couch vacuum pumps
Broke beater 125 200 Primary couch vacuum pumps
220 volts
1 Sump tank oil pumps
2 Condensate vacuum pump
3 Belt shifters
1/2 Lube oil blower
1 1/2 Suction box oscillator
2 Rectifier rolls
3/4 Consistency controllers
3 Roll wrapper
1 1/2 Reel transfer arm
1 Calender doctor oscillators
12 Bird screen drives
18 Bird screen shakers
2200 volts
1250 Synchronous drive motor
2200 volts
350 Mordens
400 Dirtec pump
2200 volts
1400 Jordans
250 Fan pump
550 volts
Lube oil pumps 10 15 1st dryer section condensate pumps
Wet broke chest agitator 10 7 1/2 Felt dryer condensate pump
Wet broke pump 7 1/2 40 Felt conditioner
White water pump 20 3 Wet strength pump
Wire suction 75 15 Filter sweetener pump
Rich white water pump 75 25 Machine chest agitator
Filtrate pump 75 40 Machine chest pump
Tailings and washup pump 20 15 Secondary beater chest pump
Fresh water pressure pump 40 25 Secondary beater chest agitator
15 Air compressor motor generator set
7 1/2 Trim blower
10 Size pumps
20 Broke beater pump
30 Calender blowers
15 2nd and 3rd dryer section condensate pumps
15 Miscellaneous condensate pumps
30 Calendar lifts
75 Hood exhaust fans
30 Heating and ventilating fans
30 Ross-Grewin air system
1 1/2 Plug roll converter
5 Size tank agitator
110/220 volts
120 Lighting
50 Jordan motor generator set
15 Cubicle room fan
10 Save-all
30 Heating and ventilating fans
2 Broke conveyer
17 Roll handling crane
50 Feeder to elevator panel

Table IV. Electrical System for No. 15 Fourdrinier



No other control instrument



has an external manual-automatic station as mistake-proof as this

***EXCLUSIVE** mechanical interlock prevents switching into wrong position*

***EXCLUSIVE** "balancing" arrangement prevents "bumping" on changeover*

Among the many features which have put the Bristol Series 500 Air-Operated Controller way out front is the unique External Manual-Automatic Station.

1. "Test" and "Service" positions are mechanically interlocked. Operator cannot, by mistake, go *through* "Manual" position into either "Test" or "Service."

2. Output pressures of the controller and the regulator on panel are measured by the *same* gauge. This enables operator to achieve exact *balance* before going from automatic to manual control or vice versa, thus eliminating the possibility of a "bump" to the process during changeover.

Bristol's External Manual-Automatic Station is an integral part of the controller installation . . . yet can be used independently for manually controlling the process *before* the controller is installed or *after* it has been removed for any reason.

Read what else Bristol Series 500 Controller gives you . . . calibrated control, single service adjustment, reset stops, etc. Write for new Bulletin A120 on Series 500 Air-Operated Controllers, THE BRISTOL COMPANY, 102 Bristol Road, Waterbury 20, Conn.

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AUTOMATIC CONTROLLING, RECORDING AND TELEMETERING INSTRUMENTS

Fig. 11. Paper test station

Instruments, left to right: fold tester, stiffness gage, burst tester, tensile tester, glarimeter, densimeter, and opacimeter.

This enables the flat box suction to be adjusted up to the capacity of the pump.

On the primary and secondary couch rolls, the vacuum is recorded, and the power to drive the helper motors is controlled (*11E*) by the vacuum carried at those points. This permits a more constant load on the belt drive and allows better control of the draw off the couch.

Press rolls are loaded pneumatically. The air pressure in diaphragm cells is regulated by valves located in the pedestals supporting each press section. Front and back trunnions are loaded separately. An instrument (*12E*) records the loadings for each press section. The same type of control is applied to the smoothing press and size press.

For control purposes, the paper and felt drier rolls are divided into five sections. The temperature in each of these is controlled by a resistance thermometer instrument (*13E*). The five controllers are grouped on a central panel for convenience of the operator. If desired, the highest temperature may be maintained in the first section of eight drier rolls in order to set the size in the paper. Each drier section has its own condensate removal system (*20E*). Thermal compressors circulate steam through the rolls and remove condensate. Siphons revolve within the paper and felt drier rolls to remove condensate which tends to follow the inner periphery as the speed approaches 1500 f.p.m. The speed of paper through the last drier section is measured by a tachometer (*5E*). This is connected with an alarm (*15E*) which warns of any sheet breakage. Total consumption and drier usage of steam are recorded by separate flowmeters(*32E*). All steam is delivered from a 150-p.s.i. system, then reduced to 100 p.s.i. at the paper machine before passing to the header supplying the drier sections.

The fourdrinier machine is driven by a line shaft connected to a single direct-current drive motor whose speed is varied by both armature and field control. The line shaft is connected to all sections of the fourdrinier through reduction gears, belts, and cone pulleys. The wire is driven by the couch rolls. The main couch is coupled with the line shaft and also to a helper motor (100 hp., direct current, 1150 r.p.m. through 6-1 gear reducer). The electrical system for the No. 15 fourdrinier is shown in Table IV. The primary couch is driven by a helper motor only (60 hp., direct current, 1750 r.p.m. through 6-1 gear reducer). The helper motors apply power directly to these rolls depending on the suction being carried. The heavier or denser the sheet, the greater will be the suction. This in turn actuates an instrument which positions a rheostat in the electrical circuit of the couch rolls to increase the power input to the helper motors. In this way, the greater the load on the wire, the more power available to drive it.

CONTROL DATA

The properties of several different grades of paper made on the No. 15 fourdrinier machine are shown in Table V. These typical grades are made from unbleached, semibleached, and fully bleached kraft and

Table V. Control Data

Paper grade kraft pulp	*Bag unbleached*	*Waxing semibleached*	*Tabulating card semibleached*	*Wet strength specialties semibleached*	*Meat wrap semibleached*	*Wrap and bag fully bleached*
Pulp brightness, % on G.E. instrument	..	30	65–67	65–67	65–67	80
Basis weight range, lb./ream of 500 24 × 36-inch sheets	30–60	40	101	45–55	42–52	35–50
Definitive Paper Tests (*16E, 17E, 30E, 35E–39E*)						
Bursting strength, lb./sq. inch/unit basis wt. × 100	81 +	91+	70+	95+	64+	53+
Aggregate tear, Elmendorf units/basis weight	4.0+	..	..	3.5+	4.0+	..
Ink, min.	..	..	..	..	30+	..
Size, sec./basis wt.	..	..	1.8	..	1–1.2	..
Caliper or thickness, inch	..	..	0.0067	..	..	..
Stiffness, taber units	..	..	18.0+ with machine direction; 8.5 + cross machine direction		..	..
Wet tensile strength, lb./0.5-inch strip/basis × 100 (with machine direction)	..	..	..	10+	..	..
Air permeability, seconds required for 100 cc. air to pass through 1 sq. inch	..	50	..	..	..	..

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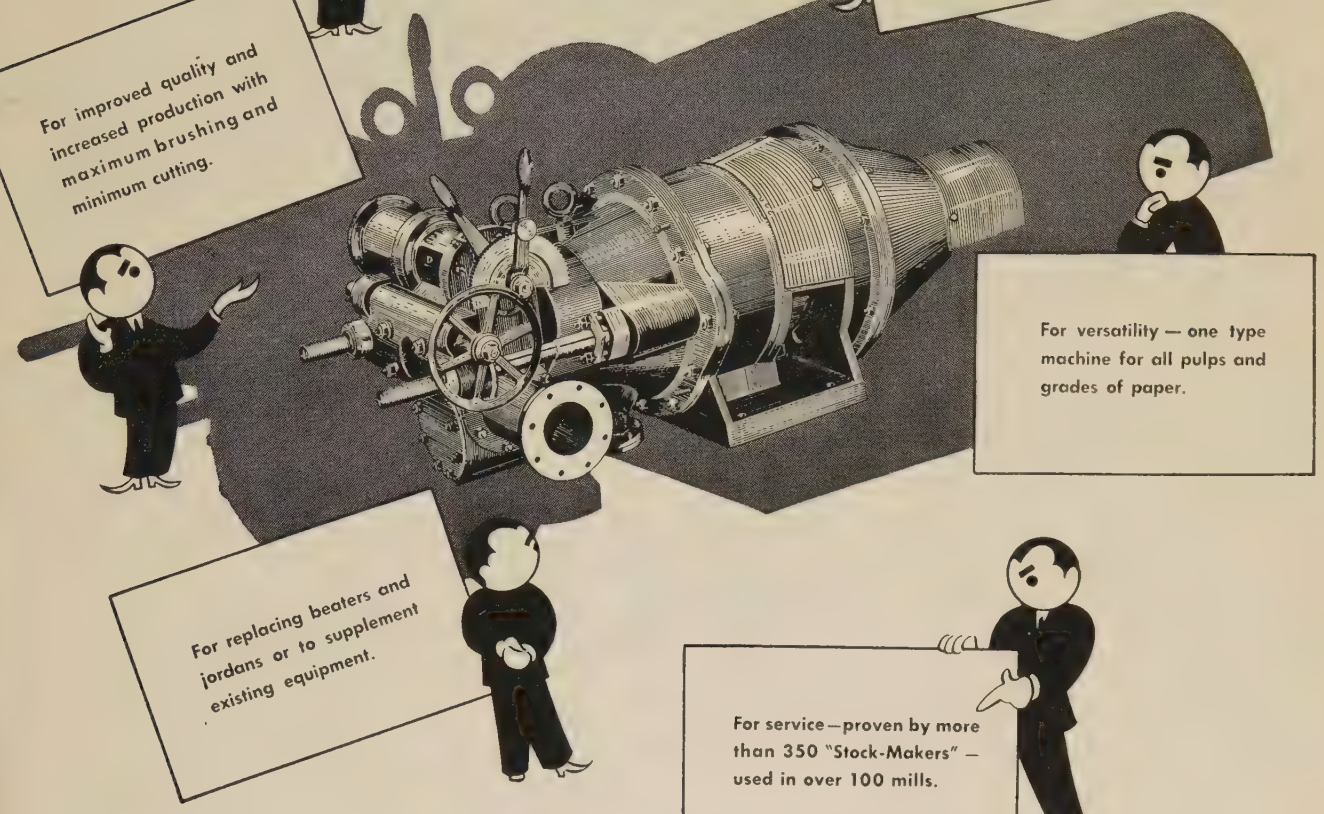

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To obtain a degree of wet strength, which is required in a number of specialties, melamine-formaldehyde resin is added at the machine stuff box. Note that this is the only item shown in Table V on which a wet tensile test is indicated.

Whatever the grade, strict adherence to the customer's requirements is ensured by quality control involving the essential paper tests such as burst (mullen), sizing, caliper, stiffness, and moisture (Fig. 10) (*8E, 16E, 17E, 30E, 35E–39E*).

FUTURE

All trends indicate that as the population of the United States increases, the per capita consumption of paper and paper products increases at an accelerated rate. Perhaps the biggest improvement in the paper-making process will be due to the application of new chemicals. Synthetic resins, synthetic rubbers, new coating compounds, more effective slime-control agents and many other specialties will all make for improved papers (*22*) in the future. Of course, some aspects of the age-old problem of sizing remain unsolved. With modifications in rosin and wax sizes, perhaps the day will come when every paper mill in the nation will look back on the dark years when sizing presented operating and specification headaches.

Just how far paper products will go in their displacement of other materials, such as metals, fabrics, and glass, is difficult to say, but many of the advances in manufacturing techniques, resulting from the stringent requirements of World War II, will be improved still further.

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2E. *Ibid.*, Fourdrinier.
3E. Bird Machine Co., South Walpole, Mass., Dirtecs.
4E. *Ibid.*, inward flow rotary screens.
5E. Bristol Co., Waterbury 91, Conn., *Bull.*, 473, Pyromaster tachometer.
6E. Brown Instrument Co., Philadelphia 44, Pa., *Bull.* 15-4R and *Cat.* 9305, Air-O-Line controller.
7E. Control Equipment Corp., Kane, Pa., *Bull.*, C-E Model B Controllers, consistency controller.
8E. Crown Williamette Paper Co., Inc., Camas, Wash., ink, size, and oil absorption tests.
9E. Fischer and Porter Co., Hatboro, Pa., Flowrator meter.
10E. Foxboro Co., Foxboro, Mass., *Bull.* M-40, proportional controller.
11E. *Ibid.*, indicating controller for press loading.
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15E. General Electric Co., Schenectady 5, N. Y., photoelectric paper break relay, G.E. no. CR 7505-K100G3.
16E. Gurley, W. & L. E., Troy, N. Y., *Bull.* 1400, densometer.
17E. *Ibid.*, *Bull.* 1441, curl and shrinkage tester.
18E. Jones & Sons Co., E. D., Pittsfield, Mass., jordan refiner (Majestic type).
19E. Jones & Sons Co., E. D., Pittsfield, Mass., beater.
20E. Midwest Fulton Machine Co., Dayton, Ohio, Fulton system.
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36E. TAPPI Standard Procedures T-412m-42, moisture test.
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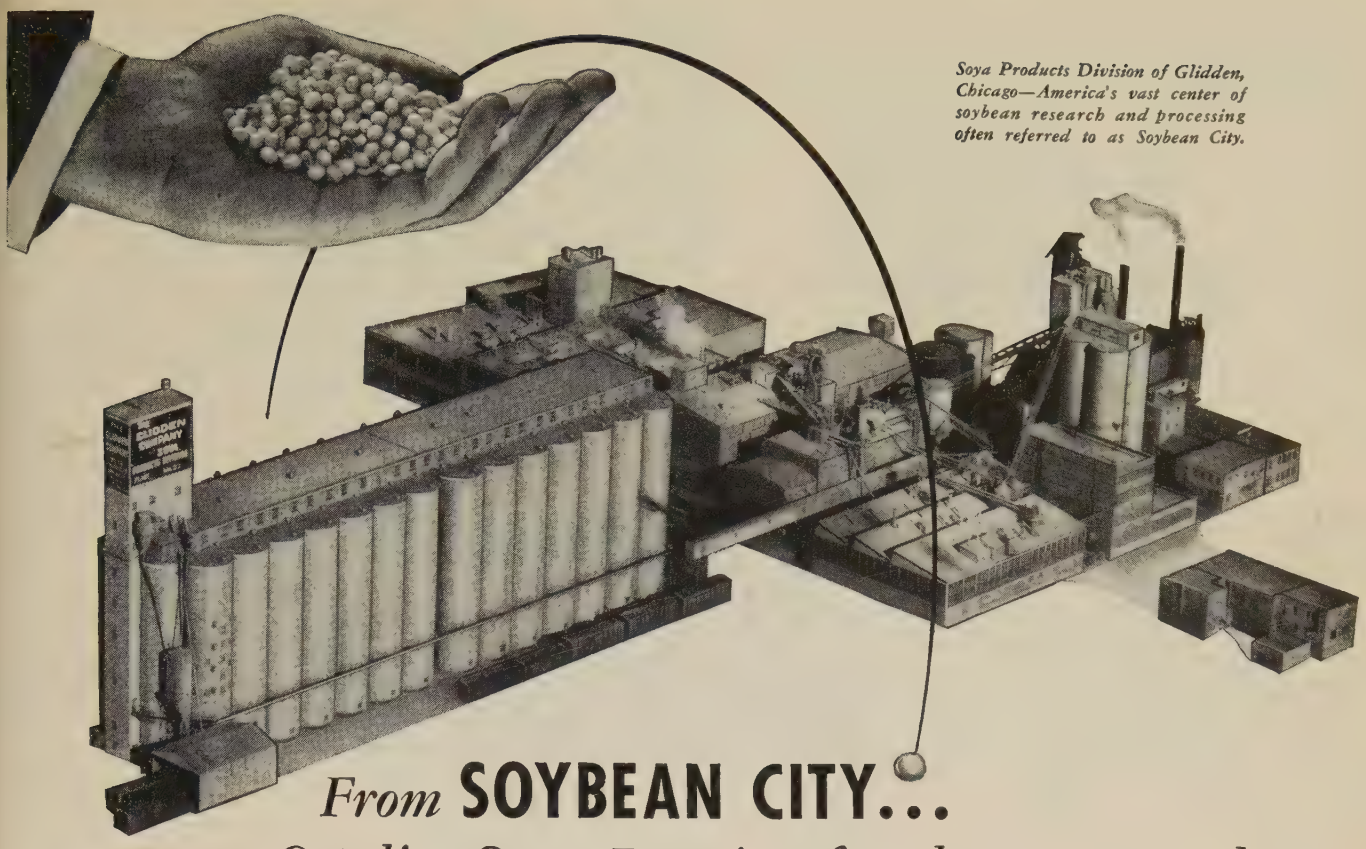

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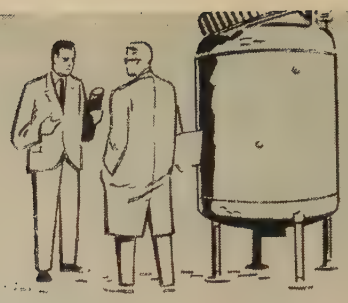

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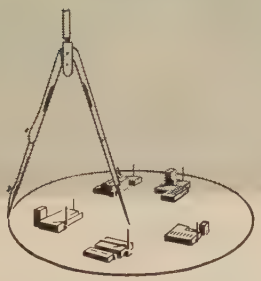

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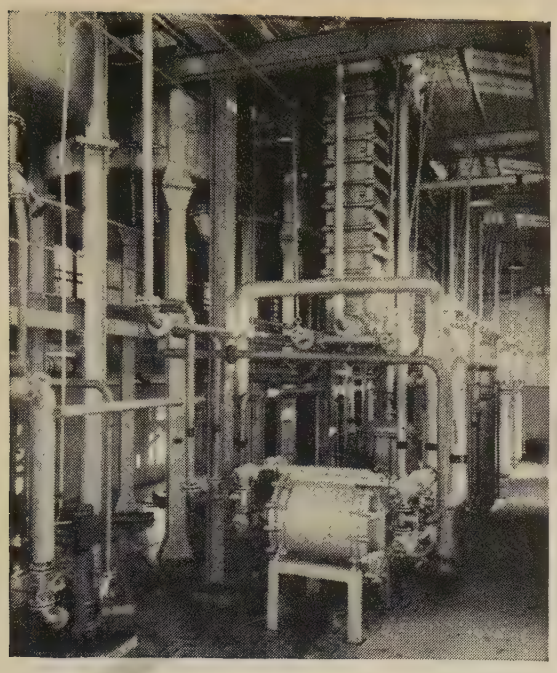

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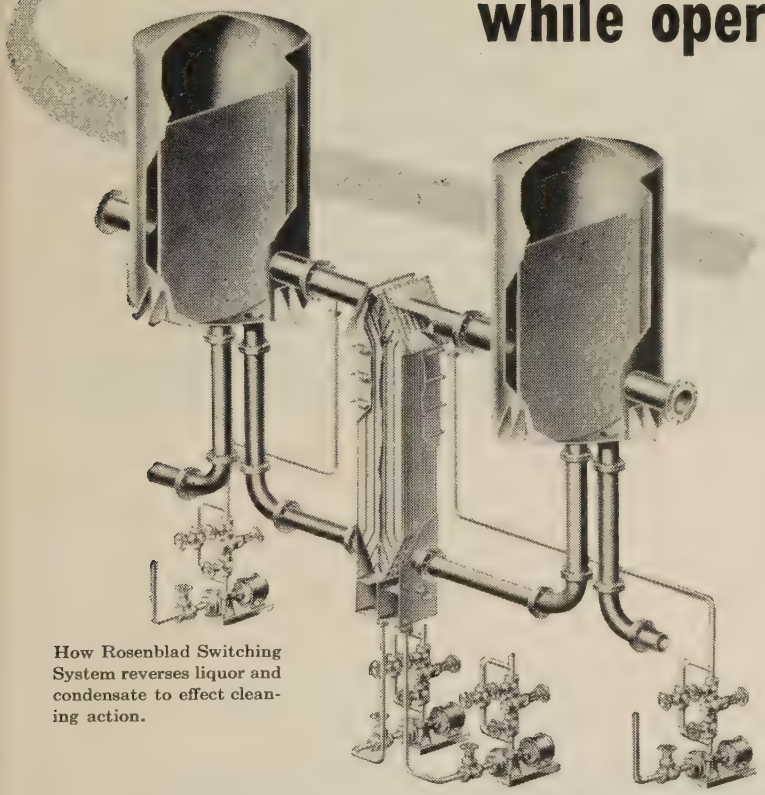

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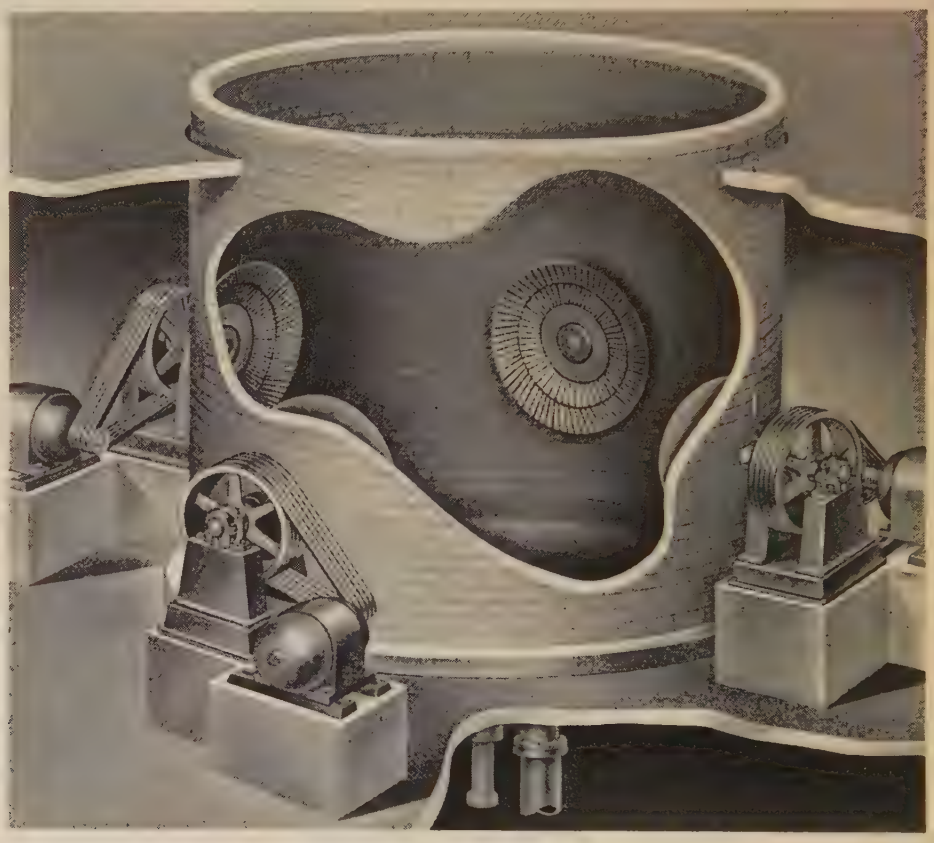

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Digester Corrosion

Experience of a Sulphate Mill

THOMAS C. JOHNSON

A study of the corrosion problems experienced in twenty digesters at the Union Bag and Paper Corp.'s Savannah plant is presented. Although the original digester service was from 12 to 14 years, newer digesters installed in 1947 and 1948 appear to be corroding so rapidly that a life of 3 to 5 years is all that can be expected. All digesters have been operated under the same service conditions. Methods for measuring rate of corrosion and inspecting digesters are discussed. The problem of digester corrosion seems to be one which cannot easily be solved by one mill and calls for considerable additional study.

THE PURPOSE of this paper is to present a summary of digester experience at the Union Bag and Paper Corp. plant in Savannah, Ga., in order to provide a basis for discussion among various sulphate pulp mills concerning the steps which can be taken to improve the life of digesters.

At this plant, there are twenty digesters which were installed at different times between 1935 and 1948. Investigations have shown that newer digesters in the same service are showing considerably greater rates of corrosion than the original equipment. All of these digesters have been purchased with exactly the same specifications for steel and code requirements.

The digesters are 9 feet 9 inches inside diameter by 41 feet $11^5/_8$ inches over-all height with typical connections for indirect cooking and liquor circulation, a 4-inch vent pipe in the neck, an 8-inch nozzle for a liquor spray ring at the top, a 10-inch liquor suction nozzle half way up the side wall with bracket supports welded to the inside wall to support the strainers, two 6-inch liquor inlet nozzles on the cone, and four 3-inch steam inlet nozzles (Fig. 1).

All digesters installed in the plant have been purchased under the same specifications for manufacture. Steel specifications were:

Table I. Specifications of Digester Steel

	Maximum percentage
Carbon	0.30
Manganese	0.80
Phosphorous	0.035
Sulphur	0.04
Copper	0.35
Tensile strength 55,000 to 65,000 p.s.i.	

The steel specified was type A.S.M.E. S-1, A.S.T.M. A-70, A.S.M.E. SA-70, or A.S.T.M. A-285. All of these numbers specify the same steel analysis, but changes in the classification system used by the A.S.T.M. and A.S.M.E. have resulted in different designations during the last few years. No coupon analyses are available for the first 15 digesters, but the analyses of the steel used in digesters 16 through 20 fall within the specifications indicated in Table I. Analyses of trepanned samples from digesters 9 and 18 by the Southern Research Institute show no significant chemical differences except in case of arsenic even though the data show a considerable variation in rate of wear of those two units (Table II). All 20 digesters were fabricated and tested in accordance with A.S.M.E. code U-68 for unfired pressure vessels for 150 p.s.i. working pressure with a corrosion allowance of $^5/_{32}$ inch. All seams were x-rayed; the entire vessels were stress relieved after fabrication; hammer tested at a hydrostatic pressure of 225 p.s.i. then raised to 300 p.s.i. and held at that pressure while all seams were inspected.

Digesters 1 through 4 have been in operation approximately 13 years, digesters 5 through 8, approximately 12 years; 9 through 13, $11^1/_2$ years; 14, 6 years; 15, $5^1/_2$ years; 16 and 17, $2^1/_4$ years; 18 through 20, $1^1/_3$ years. Typical conditions of operation of the digesters are:

Time to pressure	1 hour
Time on pressure	1 hour 20 minutes
Blowing time (approximately)	12 minutes
Maximum pressure	110 pounds (350°F.)
Type of chips	Pine (90% in the range of 1 to $^1/_8$ inch)
Total liquor volume	1150 cubic feet
Average active Na_2O in white liquor per ton of pulp	510–530 pounds
Per cent sulphidity (based on total alkali)	25–29

The first 13 digesters installed were equipped for indirect cooking. This, however, did not prove entirely successful, and the pumps and heaters were

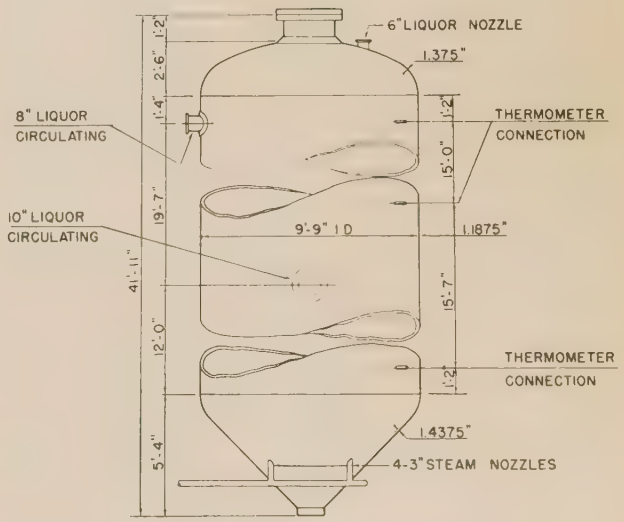

Fig. 1. Typical sulphate digester

THOMAS C. JOHNSON, Pulp Mill Supt., Union Bag and Paper Corp., Savannah, Ga.

removed in 1940. During this time, liquor circulation was used but very little indirect cooking was done. Since 1940, all digesters have been making direct cooks.

Prior to 1941, very little maintenance or inspection work was done on the digesters. During 1941, inspection of the first 13 digesters revealed that corrosion or washing-out was taking place where the steam nozzles of Type 316 stainless steel were welded

equipment and inspection procedure, we purchased the necessary instrument to make a more complete survey. The instrument which Crossett Paper Mills applied to this use is an "Audigage"—Model FMSS-5 Thickness Tester manufactured by Branson Instrument Corp. This is a portable, battery-operated unit for the nondestructive measurement of an unknown wall thickness from one side through the ultrasonic resonance principle.

Table II. Analysis of Digester Steel Samples Analyzed by Southern Research Institute 12/21/49

Per cent	*Specifications*	*Digester No. 9*	*Digester No. 18*
Carbon	0.30 (max.)	0.265	0.209
Silicon	...	0.095	0.077
Manganese	0.80 (max.)	0.46	0.50
Sulphur	0.04 (max.)	0.023	0.028
Phosphorous	0.035 (max.)	0.011	0.014
Chromium	...	0.042	Less than 0.02
Molybdenum	...	Less than 0.03	Less than 0.03
Nickel	...	Less than 0.05	0.05
Copper	0.35 (max.)	0.028	0.026
Vanadium	...	Less than 0.005	Less than 0.005
Titanium	...	0.010	Less than 0.003
Tin	...	Less than 0.001	Less than 0.001
Antimony	...	Less than 0.001	Less than 0.001
Lead	...	Less than 0.0001	Less than 0.0001
Aluminum	...	0.03	0.03
Arsenic	...	0.009	0.048

to the base metal of the digesters. These nozzles were replaced with double extra heavy steel pipe. In 1943 and 1944, extensive repairs were made to the stainless-steel lined liquor nozzles of the liquor-circulation system. During these repairs it was noted that directly under these nozzles there were washed-out places extending down the walls of the digesters for 6 or 8 feet. In the period of 1944 to 1948, extensive repairs consisted of rewelding washed-out places where the screen brackets for indirect cooking had been welded to the digester shells, replacement of liquor nozzles, and welding of larger areas on the digester shells themselves.

In 1948 the insurance underwriters would no longer permit the plant to continue the practice of building up large areas in the digester by fusion welding. Extensive welding was said to produce "locked-up" stresses which were likely to cause cracks in the shell plate. At this time, a more thorough inspection of all the digesters was started—studying thickness, points of wear, scale, pitting, grooving, etc. From results of these studies, and inspection by the underwriters, it was determined that several of the first nine digesters had reached a shell thickness which would make it necessary to replace them at an early date. It was also recommended by the underwriters, at this time, that the liquor connections no longer in use be welded up with the welds peened and ground flush with the digester walls. No other welding was permitted in the digesters without specific approval.

DETERMINATION OF DIGESTER WALL THICKNESS

Early in 1949, Crossett Paper Mills began a study of digester life which included the development of a method for measuring the wall thickness. M. J. Osborne and E. H. Jones of Crossett Paper Mills visited our plant in assembling data for their study, and in conjunction with our men, inspected three of our digesters. As a result of our observation of their

From 1941 to the present time, the methods of inspection and repairs have changed as it became necessary to do more and more repair work. During the earlier periods, inspections were made by boatswain chair, or by slowly filling the digester with chips—using the chips as a scaffold on which to work. If it were necessary to make extensive repairs to the digester, a wooden scaffold was started at the bottom and increased in height by the carpenters, permitting the boilermakers to work on different elevations of

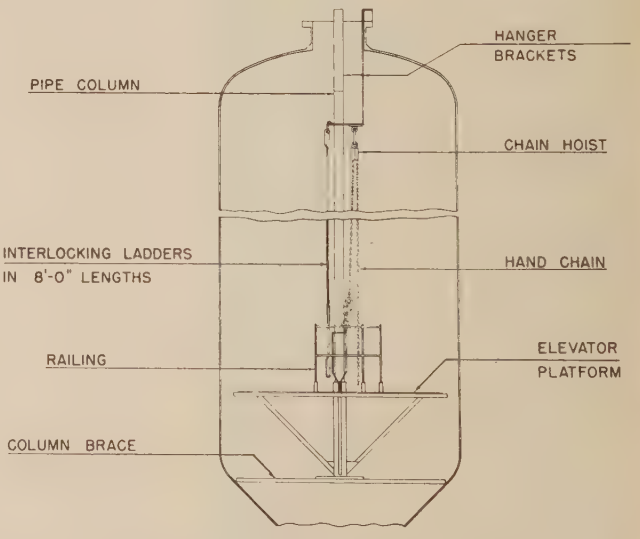

Fig. 2. Portable elevator platform for digesters

the digester. In an effort to reduce the down-time and repair costs of the digesters, the mill maintenance department developed an elevator-type scaffold which can be installed or removed from the digester in approximately 6 hours time (Fig. 2). By the use of this equipment, the men repairing the digesters are

able to work at any desired elevation without calling on other help to change their scaffold. This also made possible a visual inspection of the entire digester, and a complete audigage reading of the wall thickness in less than 8 hours.

After repairing several digesters, it was apparent that detailed records should be kept indicating the condition of the digester, repairs made, and points of wear to be carefully examined at the next inspection. The first reports were brief, and not enough information was given to keep check on the corrosion rates. Therefore, Crossett Paper Mill's method of plotting measurements showing the thickness of the walls at various points was adopted (Figs. 3 and 4). In addition, an unfolded drawing of the digester showing the condition determined by visual inspection was prepared (Fig. 5).

As a result of the inspection with the "Audigage" on all of the digesters, it was possible to determine their present condition; and by continued inspection, it is believed that it will be possible to measure accurately the rate of penetration and predict the expected life of each digester. A table has been prepared showing the rate of penetration of all of the digesters (Table III). The penetration rates of the older digesters (1 through 13), range between 0.025 and 0.040 inch with an average penetration of 0.032 inch per year. The new digesters which were placed in operation in 1947 and 1948 show penetration rates of 0.075 to 0.143 inch per year. If these latter rates of penetration continue, it will be necessary to replace these digesters after 3 to 5 years of operation, compared to 13 or 14 years in the case of the older digesters.

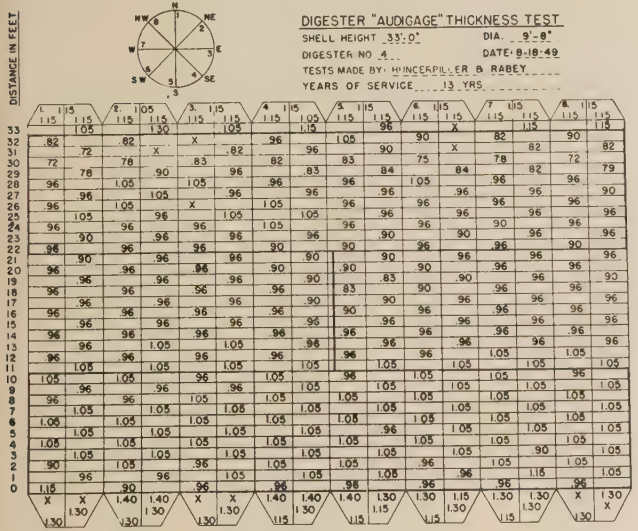

Fig. 3

It was observed in Table III that the penetration rates of digesters 5 and 9 were slightly greater than for any other of the first 13 units. Actually more accurate corrosion rates might be calculated by using penetration per 1000 cooks or per 1000 tons of pulp produced. In the case of these two digesters more pulp has been produced by them because of their physical location which permitted blowing them to either of two diffuser circles. This, therefore, resulted in their being kept in more continuous operation. However, present arrangements are such that all digesters are producing nearly the same number of

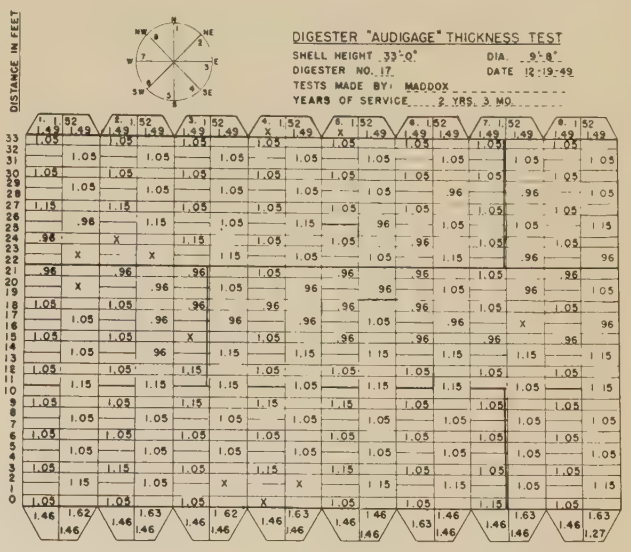

Fig. 4

blows daily. It now is considered sufficiently accurate in our particular mill to assume constant digester production and use the penetration per unit of time as a measure of wear.

Table III. Rate of Penetration of Digester Shell

Digester No.	*Age of digester, yr.*	*Original thickness, in.*	*Thickness of thinnest spot, in.*	*Penetration. in./yr.*
1	13	1.0625	0.64	0.033
2	13	1.0625	0.64	0.033
3	13	1.0625	0.68	0.029
4	13	1.0625	0.72	0.026
5	12	1.0625	0.62	0.037
6	12	1.0625	0.68	0.032
7	12	1.0625	0.72	0.029
8	12	1.0625	0.68	0.032
9	11.5	1.0625	0.60	0.040
10	11.5	1.0625	0.72	0.030
11	11.5	1.0625	0.68	0.033
12	11.5	1.0625	0.70	0.032
13	11.5	1.0625	0.78	0.025
14	6	1.0625	0.81	0.042
15	5.5	1.0625	0.82	0.044
16	2	1.150	0.96	0.091
17	2	1.150	0.96	0.091
18	1.33	1.150	1.05	0.075
19	1.33	1.160	1.06	0.075
20	1.33	1.150	0.96	0.143

REPLACEMENT EQUIPMENT

In facing the problem of selecting replacement equipment four alternatives were considered:

1. Purchase of digesters meeting the same specifications as the previous units. This would have resulted in the lowest initial cost for replacements.
2. Purchase of carbon steel digesters using steel of higher tensile strength, thus obtaining more corrosion allowance for the same shell thickness.
3. Installation of stainless-steel lined or clad digesters.
4. Installation of carbon brick lining in existing shells or new shells.

In considering carbon steel, the only alternatives seemed to be the use of A.S.T.M. A-285 steel (similar

to our present digesters) and A.S.T.M. A-212-47 steel having a tensile strength of 70,000 to 80,000 p.s.i. compared to 55,000 to 65,000 p.s.i. for A-285. The analyses and costs of these two steels are quite similar.

Table IV. Steel Specifications

	Original digesters	*Replacement digesters*	*Swedish steel*
Specification	A.S.T.M. A-285-47[a]	A.S.T.M. A-212-47	
Carbon, % max.	0.30	0.33	0.08–0.10
Manganese, max.	0.80	0.90	0.40
Phosphorous	0.035	0.035	0.03
Sulphur	0.04	0.04	0.03–0.04
Copper, max.	0.35		
Silicon	0.095[b]	0.15–0.30	0.02
Tensile strength, p.s.i.	55,000–65,000	70,000–80,000	. . . .

[a] Also specified as A.S.T.M. A-70 and A.S.M.E. SA-70.
[b] Based on analysis by Southern Research Institute.

This table shows an analysis of Swedish steel which was reported to give good corrosion resistance in digester service. Use of this steel was considered, but the digester manufacturers reported that this was "rim steel" which was unacceptable for use in pressure vessels fabricated to meet code and insurance company requirements.

A cost comparison developed that more corrosion allowance could be obtained using A-212 steel and thicker plate at a very slight additional expenditure. Three digesters are currently being installed using Type A.S.T.M. A-212 steel $1^3/_8$ inches thick in the upper 8-foot section and $1^3/_{16}$ inches thick in the lower section, resulting in corrosion allowances of $^{21}/_{32}$ and $^{15}/_{32}$ inch, respectively, compared to $^5/_{32}$ inch in the original digesters.

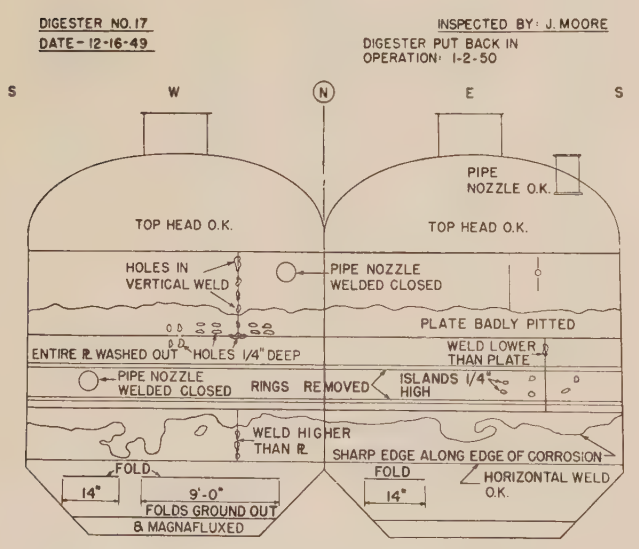

Fig. 5

Consideration of carbon brick linings was deferred, pending further investigation of installation already in operation and development of more complete cost comparisons. Our first studies assumed the use of 5 inch thick carbon lining which reduced the digester volume to a point where additional digesters were required. Subsequently, the manufacturer recommended 3 inch thick lining and it was ascertained that digesters of 6 inches greater diameter could be installed in place of the original digesters. Under these conditions cost comparisons show carbon brick lined digesters to be worthy of serious consideration.

At the time of our first consideration of stainless-steel clad and stainless-steel lined vessels several installations using various types of stainless steel were in progress. Therefore purchase of this type of construction was deferred until more information concerning proper alloy and type of construction was available At this particular time, we were not entirely aware of the extremely rapid corrosion rate being experienced in the newest digesters; and, therefore, were comparing costs on the basis of the 12- to 15-years life then indicated in the original digesters.

Considerations for the next group of replacements are being made based on more complete knowledge of the condition of all twenty of our digesters. It now appears that we must make our comparison on a life expectancy not exceeding 5 years for carbon steel digesters. The installed costs of the three types of digesters we are considering are: (1) Carbon steel, $23,000; (2) Carbon brick lined steel $31,000; and (3) Type 316 Cb stainless clad steel $44,000. With these costs it is only necessary to obtain approximately 7-years life from carbon brick lined digesters, or approximately 10-years life from stainless-steel clad digesters to have comparable annual installed cost. In these cases, it has been assumed that maintenance costs per year would be approximately equal for each of the three types under consideration.

SUMMARY

At the Union Bag plant in Savannah, digesters installed in 1947 and 1948 are showing corrosion rates two and one-half to five times as great as digesters installed in 1936 and 1937. All of these digesters were fabricated from steel meeting similar general specifications and have been operated under the same conditions of service. A digester life of 12 to 13 years was considered sufficient to justify use of low-carbon steel for replacement because of price consideration and lack of definite information concerning life of alloys and brick linings. However, a life of 3 to 5 years using carbon steel is so short that the use of alloys or carbon brick lining appears absolutely necessary.

Recent estimates indicate that carbon brick lining can be justified if it will last 7 years compared to 5 years for carbon steel. Stainless-steel clad digesters can be justified if they will last 10 years. Observations indicate that the rapid wear must result from some change which has taken place in the steel composition or in methods of fabrication. This presents an important problem which cannot easily be studied by individual mills. In the author's plant, it has been found desirable to set up a very complete inspection system to enable us to keep the digesters in proper repair and to predict the proper time for replacement so that changes may be made in an orderly manner without loss of production.

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Quality Control Charts with Constant Limits for Subgroups of Different Size

J. F. LANGMAID, JR. and H. W. COBB

In the use of Shewhart control charts, the best choice of rational subgroups sometimes leads to subgroups of different size. The usual construction of control charts then displays wider limits for small groups than for large ones. This can be a source of confusion in reading the chart, because the statistical meaning of the limits is the same regardless of their width. To avoid confusion a simple transformation has been devised which eliminates the influence of subgroup size on the location of limits but leaves their meaning unchanged.

THE Shewhart control chart is one of several statistical devices used in industry to reveal the presence of abnormal quality and process variation. In essence, the method depends on marking off "rational subgroups" of test results or other observations. These subgroups are selected in such a way that the variability within each one may be presumed to result only from chance causes inherent in the process. Then one determines whether the variation among subgroups is greater than can be statistically accounted for on the basis of the variability within subgroups. The success of the method depends on the proper selection of the subgroups.

At S. D. Warren control charts are used to follow the operation of one of the largest paper machines. On this machine several different grades and basis weights are made, and an entire "run" of paper defined by the furnish and basis weight was chosen for the rational subgroup. Unfortunately, the runs differ in length with the result that conventional control charts, when constructed for these runs, are confusing in appearance, as will be explained. However, by slightly modifying the standard construction of the charts, it was possible to simplify their appearance and make their interpretation easier.

The symbols used in the following paragraphs are the same as used in the ASTM Manual on Presentation of Data (*1*). Thus:

- $\bar{X}$ = average value within a subgroup, an estimate of $\bar{X}'$.
- $\bar{X}'$ = standard value, or expected value of average.
- s = standard deviation of a subgroup.
- c_2 = factor correcting for sampling bias in standard deviation.
- s' = universe standard deviation.
- n = number of observations in a subgroup.

In the "classical" method of plotting control charts for $\bar{X}$ where the size of the rational subgroups differs from one subgroup to another, the control limits are given by $\bar{X}' \pm ts'/\sqrt{n}$, where t is set at some economic value, usually 2 or 3. Because of the factor $\sqrt{n}$ in the denominator, the control limits change with subgroup size. The resulting chart must be interpreted in terms of the distance of each point from the central line, relative to the distance of the control limits from the central line. For example, two points can be at the same absolute distance from the central line and yet one may be in control while the other is out of control. This is a disadvantage in some uses of control charts, e.g., where they are used in quality reports to management. For such uses, the meaning of the chart should be immediately apparent, and the distance of each point should directly indicate the degree of control achieved, and whether it is satisfactory or unsatisfactory.

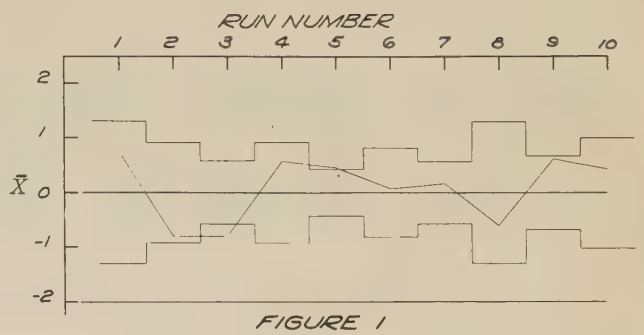

FIGURE 1

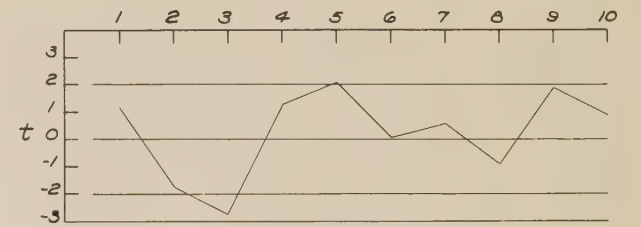

FIGURE 2

In order to show directly on the chart the relative deviation from the central line rather than the actual deviation, we plot not the actual average $\bar{X}$ of the subgroup but $t = (\bar{X} - \bar{X}') \div s'/\sqrt{n}$. This is the ratio of the actual deviation to the standard deviation for subgroups of the given size. Control limits placed at $t = \pm 2$ correspond to two-sigma limits on the conventional type of chart.

The same idea can be used for control charts on variability. However, the expected value of s depends on subgroup size because of the bias in its sampling

Table I. Difference Between Actual and Standard Basis Weight. Universe Standard Deviation $s' = 1.45$

Run No.	n	$\bar{X}$	$s'/\sqrt{}$	t
1	5	0.75	0.65	1.16
2	10	−0.80	0.46	−1.75
3	25	−0.80	0.29	−2.76
4	10	0.58	0.46	1.27
5	46	0.44	0.21	2.05
6	12	0.06	0.41	0.01
7	27	0.16	0.28	0.57
8	5	−0.60	0.65	−0.93
9	18	0.64	0.34	1.88
10	8	0.44	0.51	0.86

J. F. LANGMAID, JR., Member TAPPI; and H. W. COBB, S. D. Warren Co., Cumberland Mills, Me.

distribution. Instead of correcting each central line for bias, it is more convenient to use s/c_2 in place of s. The expected value of s/c_2 is s', and thus the central line is independent of subgroup size. Then, by analogy with the method used for averages, $(s/c_2 - s') \div s'/c_2\sqrt{2n}$, with limits at ± 2 is plotted.

As an example, consider the construction of a control chart for the data in Table I which represents the average difference between actual basis weight and standard basis weight in successive runs of paper. Figure 1 shows the standard chart on which are plotted the values $\bar{X}$ with control limits at $\bar{X}' \pm 2s'/\sqrt{n}$. The last column in the table gives t, the ratio between $\bar{X}$ and $s'/\sqrt{n}$. These values are plotted in Fig. 2, with limits at ± 2. It is apparent that Fig. 2 displays the same essential information as Fig. 1 but in a form which is more readily grasped.

LITERATURE CITED

1. American Society for Testing Materials, "ASTM Manual on Presentation of Data," Philadelphia. Reprinted 1949.

Received May 9, 1950.

Relations Between Yield and Properties of Spruce and Aspen Sulphite and Sulphite Semichemical Pulps

J. N. McGOVERN

Relations between the yield of pulp and its strength properties and chemical composition were determined for black spruce (*Picea mariana*) and quaking aspen (*Populus tremuloides*) sulphite pulps ranging in yield from that of full chemical to that of semichemical processing. Optimum pulp strength was obtained at yields near 50% and good strength relative to the optimum was found at yields near 60%. Pulps made in yields of approximately 75% were weak and bulky in comparison with pulps made in lower yields. The lignin and pentosan content of the pulps and their permanganate numbers increased and cellulose content decreased with increasing yield.

The sulphite pulping industry has always been confronted with the problems of improving the yields of pulp obtained within the ranges established by current grades at the time and extending the limits of the ranges beyond those of current usage. Past efforts to find solutions to these problems, however, have apparently been inadequate to meet the present needs raised by higher wood costs and diminishing supplies of favored pulpwoods. There are gaps in the information needed to obtain the desired results. A particular gap seems to lie between the known information on the relations of yield to properties and bleachability of the practical restricted range of sulphite pulping and the academic knowledge obtained for the most part from work with wood meal, covering the full range of yields but related almost entirely to chemical constituents.

The purpose of this study was to help fill in this void by determining the properties of spruce and aspen pulps made over a wide yield range, from that of full chemical to that of semichemical pulps. Although pulp yield and properties can be changed by varying the conditions of time, temperature, and acid concentration employed, digestion time was made the main variable in this study, with minor consideration given to acid concentration. For the latter purpose, acids of high and low concentration within the limits of commercial practice were used. Other aspects of high-yield pulping, such as blowing the digester, fiberizing and refining, screening, bleaching, and removing dirt, were not considered in this investigation, although all these factors are highly important in this kind of pulping.

Several surveys and reviews have covered the factors in the relations between sulphite pulp yield and properties and have shown the great interest and possibilities in this field (*2, 7, 9, 10*).

EXPERIMENTAL PART

The sulphite digestions were made on $^5/_8$-inch chips from Canadian black spruce and Wisconsin quaking aspen. The spruce digestions were made in a stainless-steel-clad, tumbling autoclave equipped with a steam jacket and having a capacity of 1.5 cubic feet. The aspen digestions were made in a stainless-steel-lined digester equipped with a steam jacket and having a capacity of 13 cubic feet. Most of the digestions were made in duplicate; the exceptions are noted in Table I. The autoclave digestions were discharged by dumping. Those made in the larger digester were discharged by blowing for the lower-yield pulping and by dumping for the higher-yield pulping.

The sulphite cooking acid was prepared by passing sulphur dioxide from a cylinder into lead-lined tanks containing milk of lime. Fresh acid was used for each digestion.

The average moisture content of the spruce chips was 12% and that of the aspen chips 32%. The other digestion conditions are given in Table I.

After dumping the digested spruce chips from the autoclave, the handling procedure varied somewhat for the different series with the low- and high-concentration acids. The material from the high-concentration digestions was placed in a tank equipped with a small stirrer. The contents were circulated for 5 minutes, emptied into a screen box, and screened through a diaphragm screen with 0.012-inch slots. The screening rejects for the higher-yield pulps were disintegrated in a 5-pound beater and rescreened before the accepted pulp was tested. The material from the low-concentration digestions was stirred, screened, and disintegrated in this way only for the lower-yield digestions; the

J. N. McGovern, Chemical Engineer, Forest Products Laboratory, maintained at Madison, Wis., in cooperation with the University of Wisconsin, Forest Service, U. S. Department of Agriculture.

Table I. Digestion Conditions and Pulp Yields for Sulphite Digestions of Quaking Aspen and Black Spruce

Digestion No.	Acid composition Total SO_2, %	Acid composition Combined SO_2, %	Maximum temperature, °C.	Maximum pressure, p.s.i.	Total time, hr.	Combined SO_2 at end, %	Total pulp yield, %	Screenings, %	Permanganate	Pulp brightness, %
Aspen digestions[a]										
5187[b]	5.55	1.08	148	90	6.4	0.14	47.7	..	11.9	42
5188[b]	5.60	1.11	141	90	5.8	..	52.8	..	21.0	36
5189–92	5.50	1.10	136	90	5.0	0.51	69.4	..	37.0	38
5190–93	5.50	1.10	130	90	4.5	0.61	76.3	..	..	39
5191[b]	5.50	1.10	122	90	4.0	0.81	89.4	..	..	43
Spruce digestions with low acid concentration[c]										
421–7	4.06	1.00	130	79	14.0	0.03	49.4	0.8	9.4	60
420–8	4.00	1.00	130	78	12.0	0.13	52.4	1.0	17.9	52
422–9	4.02	1.00	130	77	10.5	0.22	55.8	1.2	28.6	49
423–4	4.03	1.00	130	72	9.0	0.24	63.3	..	..	37
425–6	4.01	1.00	130	71	7.5	0.38	70.6	..	..	37
Spruce digestions with high acid concentration[c]										
397[b]	5.97	1.30	130	80	12.0	..	49.3	0.1	10.7	56
394[b]	5.96	1.31	130	80	11.0	0.26	51.3	0.9	14.9	56
395–8	5.99	1.32	130	80	9.0	0.31	56.4	1.4	24.2	45
403–4	6.05	1.29	130	80	8.2	..	61.6	18.0	41.5	46
399–02	6.02	1.30	130	80	7.5	0.50	63.4	51.0	43.6	41
401[b]	5.90	1.33	130	76	6.0	0.64	75.0	..	..	39

[a] (1) The temperature schedule was: 0.5 hour from 38 to 90°C. using indirect steam; 0.5 hour at 90°C., 2 hours to 104°C., 2 hours to 136°C., and 1.5 hours to 146°C. using direct steam (shorter digestions terminated at intermediate points).
(2) The liquor-to-wood ratio as charged was 62.5 gallons of cooking acid per 100 pounds of moisture-free wood.
[b] Single digestions; others duplicate digestions.
[c] The chips were presteamed for 0.5 hour at atmospheric pressure. The temperature schedule was: 0.5 hour from 30 to 90°C., 2.5 hours at 90°C., 3 hours to 130°C. and hold at 130°C. for digestion duration, using indirect steam. The liquor-to-wood ratio as charged was 55 gallons of cooking acid for 100 pounds of moisture-free wood.

higher-yield digestions were fiberized first in the 5-pound beater and then screened. All of the aspen pulps or partially pulped aspen chips were fiberized in a commercial, double-disk attrition mill to a freeness of approximately 700 ml. (Schopper-Riegler) and screened through 0.008-inch screen plates.

The pulps were tested for strength and chemical composition by standard TAPPI methods.

The aspen pulps were blended with commercial unbleached spruce and bleached aspen sulphite pulps in a fixed proportion, and the mixtures were made into standard test sheets and tested for strength by the standard methods. The experimental pulps used in the mixtures were not given processing other than the treatment received in the first fiberizing, and the commercial pulps were used as received.

DISCUSSION

Relation of Digestion Conditions to Pulp Yield

The relations between total pulp yield and digestion time for the spruce and aspen pulping shown in Fig. 1 were typical for the sulphite pulping of wood (*1, 4, 10*). The effect of acid concentration is also shown in Fig. 1. The rate of pulping was faster with the high than the low concentration for the same delignification. This difference was probably due to the high concentration of free sulphur dioxide in the high-concentration acid (*10*). In addition to increasing the rate of pulping, the high sulphur dioxide concentration probably aided in penetration of the sulphite acid into the chips. This is important because uniformity and completeness of penetration are particularly necessary in high-yield pulping.

Table II. Physical Properties and Chemical Composition of Quaking Aspen and Black Spruce Sulphite Pulps

		Pulp physical properties based on ream of 500 sheets each 25 by 40 inches												Pulp chemical composition			
		Bursting strength Freeness[a]		Tearing strength Freeness[a]		Tensile strength Freeness[a]		Folding strength Freeness[a]		Test sheet density Freeness[a]		Beating time Freeness[a]					
Digestion No.	Total pulp yield, %	800 ml., pt./lb.	550 ml., pt./lb.	800 ml., g./lb.	550 ml., g./lb.	800 ml., m.	550 ml., m.	800 ml., double folds	550 ml., double folds	800 ml., g./ml.	550 ml., g./ml.	800 ml., min.	550 ml., min.	Lignin %	Cellulose, %	Alpha cellulose, %	Total pentosans, %
Aspen digestions																	
5187	47.7	0.46[b]	0.54	0.54	0.45	4700	5400	13	53	0.70	0.78	6	66	2.7	94.8[c]	83.6	6.5
5188	52.8	0.60[b]	0.64	0.52	0.35	5150	5750	33	120	0.74	0.80	56	87	6.9	88.2	74.6	13.4
5189–92	69.4	0.40[b]	0.48	0.46	0.45	3900	4400	10	30	0.63	0.68	0	22	11.4	77.7	64.9	10.9
5190–93	76.3	0.24[b]	0.30	0.36	0.40	2750	3150	3	6	0.52	0.56	3	31	12.4	72.7	59.5	13.2
5191	89.4	0.04[b]	0.18	0.20	0.20	1550	1840	0	0	0.39	0.43	4	31	12.6	73.6	56.1	18.0
Spruce digestions with low acid concentration																	
421–7	49.4	0.97	1.28	1.37	1.04	6550	8750	645	935	0.74	0.90	12	39	0.8	95.7[d]	79.5	5.0
420–8	52.4	1.04	1.32	1.30	1.00	7350	8900	660	985	0.78	0.89	12	35	2.0	92.4	77.0	5.9
422–9	55.8	1.10	1.28	1.16	0.96	7450	9250	440	600	0.74	0.83	11	33	6.4	86.6	71.9	5.8
423–4	63.3	0.97	1.12	0.95	0.74	6650	7850	255	280	0.61	0.70	15	34	13.6	74.5	62.0	6.3
425–6	70.6	0.66	0.72	0.92	0.72	4800	6500	100	102	0.45	0.52	24	44	20.0	70.8	58.5	6.6
Spruce digestions with high acid concentration																	
397	49.3	0.96	1.11	1.15	0.90	7100	8200	420	485	0.74	0.87	11	33	0.6	94.7[d]	76.2	5.5
394	51.3	0.90	1.18	1.15	0.82	6450	8350	200	580	0.76	0.88	11	34	1.5	93.6	75.4	5.9
395–8	56.4	0.97	1.08	0.98	0.75	6500	8500	310	360	0.70	0.80	18	41	5.4	87.0	70.6	5.9
403–4	61.6	0.80	1.06	0.92	0.70	6250	7400	250	330	0.60	0.72	12	35	10.8	79.9	66.4	6.8
399–02	63.4	0.82	0.94	0.94	0.68	6000	7300	150	175	0.58	0.66	14	34	13.4	76.9	62.0	6.5
401	75.0	0.51	0.59	0.95	..	3400	5000	50	50	0.42	0.51	32	54	21.9	66.8	51.6	7.5

[a] Schopper-Riegler freeness.
[b] 650-ml. freeness.
[c] Holocellulose.
[d] Cross and Bevan cellulose.

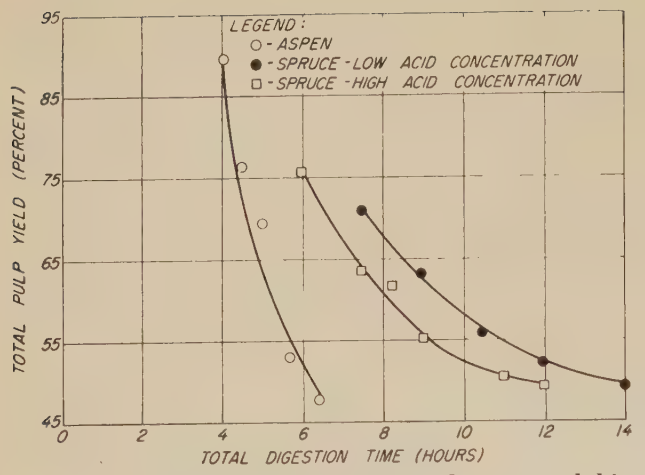

Fig. 1. Relations between spruce and aspen sulphite pulp yields and digestion time

Relation of Pulp Yield to Screenings and Permanganate Number

The yield of screened pulp, which was investigated with the high-concentration spruce pulping, reached a maximum at about 55% and then dropped off sharply (Fig. 2). This yield corresponded to a lignin content of about 5%. With less delignification, the fibers did not separate readily with the mild stirring given the pulped chips after they were discharged from the autoclave. The yield value for this fiberizing point would probably be different in commercial practice where blowing is employed and chip size and pulping conditions are less uniform.

The permanganate number decreased with increasing pulping time and paralleled pulp yield in this respect (Table I and Fig. 2). The spruce pulps made with both high and low acid concentration showed a coinciding relationship between permanganate number and pulp yield (Fig. 3). Similar relations of pulp yield to permanganate number and lignin content are evident in Fig. 3.

Relation of Pulp Yield to Strength

The bursting and tensile strengths of pulps beaten to the same freeness were found to be maximum at yields near 55%, whereas in general the tearing and folding strengths decreased continuously with increasing yield through the whole yield range (Table II and Fig. 4). The trends for yields up to about 60% are in general agreement with the results of other investigators (*3, 8*). The density of the test sheets, however, also passed through a maximum at yields near 55% and decreased appreciably with increasing yield above that point (Fig. 4). This complicated the analysis of the effect of pulp yield on pulp strength because it was not possible to compare pulps with the same fibrillation, as measured by freeness, and the same density of pulp test sheets.

Part of the variation in density values was caused by the error in making thickness measurements on the rough surfaced test sheets obtained from the high-yield pulps. This had a tendency to produce lower values than normal. On the other hand, the freeness values of the high-yield pulps are likely to have been lower than if caused only by the fibrillation produced in beating, because of the possible production of fines in the previous fiberizing operation. Yet, in spite of these obscuring effects, it is evident that the pulps made in yields higher than 65% were appreciably lower in strength than those made in yields of 50 to 55%. This was especially true for folding strength which may be more sensitive than the other strength properties to variations in fiber bonding and fiber flexibility. It is of considerable interest to note that the pulps made in yields near 60% were only slightly lower than the maximums in strength.

Table III. Strength Properties of Test Sheets Containing Experimental Aspen Sulphite and Sulphite Semichemical Pulps[a]

Digestion No.	Pulp yield, %	Strength properties[b] Bursting strength, pt./lb.	Tearing strength, g./lb.	Tensile strength, m.	Test sheet density, g./ml.	Pulp freeness (Schopper-Riegler), ml.
5188	52.8	0.40	0.85	3720	0.65	805
5189–92	69.4	0.37	0.85	3900	0.65	770
5190–93	76.3	0.31	0.83	3050	0.61	790
5191	89.4	0.27	0.80	2570	0.61	795

[a] Thirty-five per cent experimental aspen pulp (digestion as indicated), 35% unbleached spruce sulphite pulp (shipment No. 2427), and 30% bleached aspen sulphite pulp (shipment No. 2428).
[b] Based on 25 × 40—500.

The difficulty of evaluating the high-yield aspen pulps was minimized greatly by blending them with commercial unbleached spruce sulphite and bleached aspen sulphite pulps (Table III). The bursting and tensile strengths of the pulp mixtures decreased in proportion to the strength of the experimental pulp present and the tearing strength somewhat less, although the freeness and density values were nearly the same. These results verified the observation on the decrease in pulp strength with increasing yield.

The spruce pulps made with the cooking acid of low concentration were stronger than those made with the high-concentration acid for yields up to 65% (Table II and Fig. 4). The difference was greatest for folding strength (Table II). At the highest yields, the two types of pulp were the same in all except folding strength. Apparently the slower pulping with the low-concentration acid gave pulps with better bonding characteristics than the faster pulping with the stronger acid. This trend, especially with respect to sulphur dioxide concentration, has been observed in other research (*5, 6*).

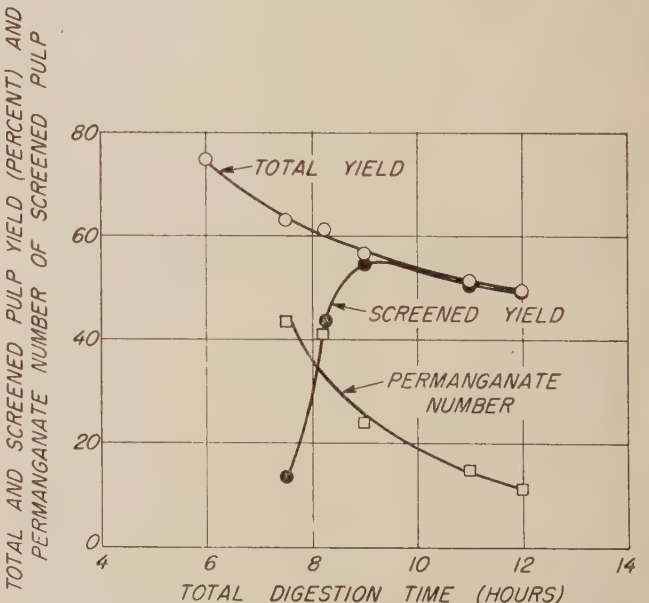

Fig. 2. Relations between spruce sulphite pulp yield, screenings, and permanganate number and digestion time. (Acid concentration, 6.0% total with 1.3% combined sulphur dioxide.)

The spruce pulps were characteristically stronger than the aspen pulps. For a given yield, the aspen pulp had roughly one half the strength of the spruce. The spruce pulp of lowest strength was as strong as the strongest aspen pulp. Part of this difference may have been due to the relatively high digestion temperatures used for the aspen pulping, but the greatest part of the difference is probably explained by the shortness and poorer bonding characteristics of the aspen sulphite fiber.

Relation of Pulp Yield to Chemical Composition

The pulping reactions gave increasing lignin, decreasing cellulose, and increasing pentosan contents with increasing pulp yield (Table II and Fig. 3). The cellulose and lignin relations were practically linear for the practical yield range of 50 to 75%, except for the aspen lignin-yield relation which showed a lower rate of increase above than below a pulp yield of 60%. The aspen pulps made in yields below about 60% had higher lignin and lower cellulose content relative to those from spruce than would be expected from the relative compositions of the woods. Above this yield the relations agreed with expectation. The pentosan content of the spruce pulps increased slightly with pulp yield (Table II), but an appreciable proportion of this constituent was removed before a yield of 70 to 75% was reached, as shown by the relatively low pentosan contents of the highest yield pulps. The aspen pulps showed a wider range of pentosan content and higher figures for a given pulp yield than the spruce pulps because of the high initial pentosan content of the aspen wood.

The chemical composition of the spruce pulps was not affected by the cooking-acid concentration (Fig. 3). It is possible that, in producing pulps of the same lignin content under the two acid concentrations, the normally opposite effects on lignin removal caused by decreasing both the free sulphur dioxide and the calcium bisulphite were balanced. The lack of effect of changing acid concentration on chemical composition of the pulps did not reflect the differences found in pulp strength.

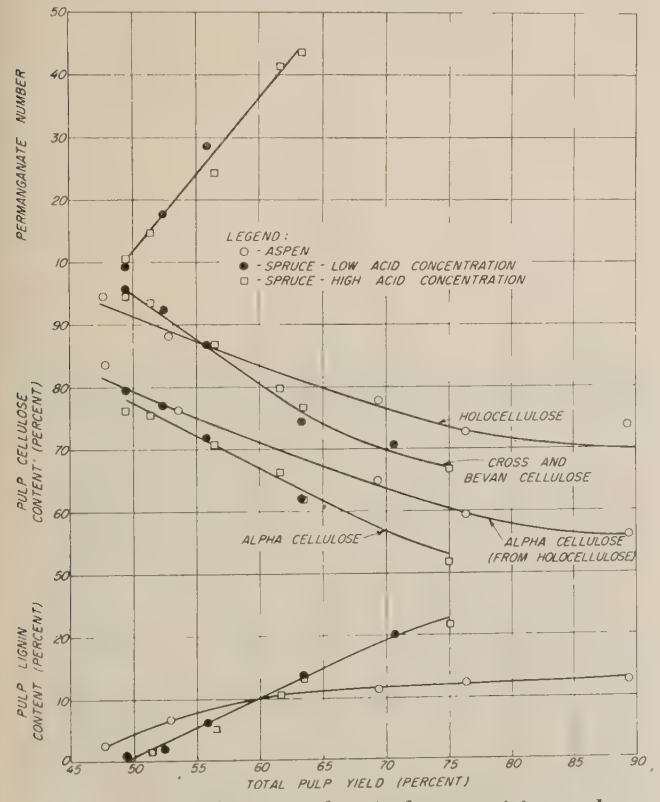

Fig. 3. Relations between chemical composition and permanganate number of spruce and aspen sulphite pulps and pulp yield

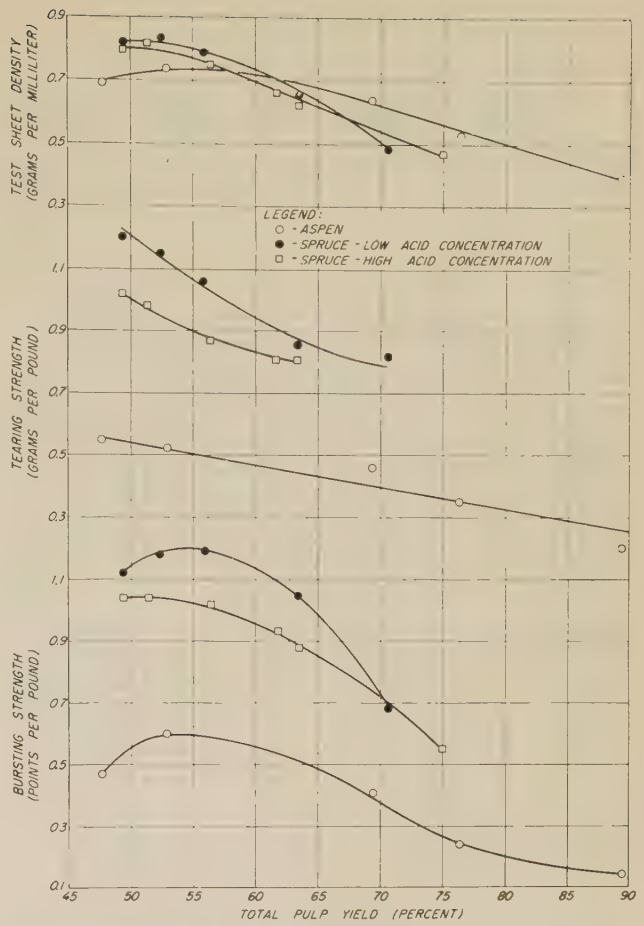
TEST SHEET DENSITY (GRAMS PER MILLILITER)
TEARING STRENGTH (GRAMS PER POUND)
BURSTING STRENGTH (POINTS PER POUND)
TOTAL PULP YIELD (PERCENT)
LEGEND:
○ - ASPEN
● - SPRUCE - LOW ACID CONCENTRATION
□ - SPRUCE - HIGH ACID CONCENTRATION

Fig. 4. Relations between physical properties of spruce and aspen sulphite pulps and pulp yield. (Average of values at 550 and 800 ml. Schopper-Riegler freeness.)

Relation of Pulp Yield to Brightness

The spruce pulps showed decreasing brightness with increasing yield (Table II). All the aspen pulps were low in brightness for no apparent reason and, unlike the spruce pulps, showed no trend with increasing yield.

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The Effect of Roll Load in the Beating of Southern Kraft Pulp

C. S. WALSETH and C. G. GEIJER

Four 2000-pound Jones-Bertrams beaters are included in the stock preparation system of No. 5 machine at the Savannah plant of the Union Bag and Paper Corp. A brief description of this installation is given. In an effort to increase the refining capacity of these beaters, a study was made of the effect of increased roll load on capacity, quality, and power consumption. Variation in roll load is accomplished by means of pneumatic cylinders, and loads up to 200% of roll weight were studied. Special beater runs were arranged during the course of regular production, and stock samples taken at intervals were evaluated by freeness measurements and physical tests on TAPPI standard handsheets. The pulp was unbleached kraft with a permanganate number of about 26. The results indicate that increase of roll load up to 200% for beating times up to 30 minutes will increase refining capacity, but for longer beating times the advantage of roll loads above 125% is lost. The same degree of strength development is obtained with less power consumption as roll load is increased, except at high roll loads and long beating times. The relationship between bursting strength and tearing strength showed that some loss in tear results from increasing the roll load from 125 to 200%.

At the Savannah plant of Union Bag and Paper Corp., the stock preparation system for No. 5 machine includes four 2000-pound Jones-Bertrams beaters. These beaters are part of a flexible stock preparation system which was designed to accommodate the production of specialty grades utilizing either dry, lap, or slush pulp. If dry or lap pulp is used, it is defibered in a Hydrapulper which discharges to a stock chest from which the beaters are supplied. This Hydrapulper chest may also be supplied with slush pulp from a decker chest. After the beating cycle, the stock from each beater may be dumped to either of two beater chests. Stock may be transferred from one beater chest to the other, or it may be pumped from either beater chest to four jordan refiners. The stock flows from the jordans directly to the paper-machine regulator box. The use of two beater chests makes possible sharp change-overs in type of stock or furnish.

Under present conditions, No. 5 machine is normally operated on regular grades of unbleached kraft bag papers, particularly heavier weights. Since production rates on these grades are unusually high, the stock may be held in the beaters for only a short time and it is thus desirable to obtain the maximum refining capacity from a short beating cycle. Thus, an experimental study was undertaken with the objective of increasing the refining capacity of these beaters. In the course of this investigation, a study of the effect of roll load on capacity, quality, and power consumption was made. It was believed that the results of this study would be of general interest.

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DESCRIPTION OF BEATER INSTALLATION

The four Jones-Bertrams beaters are each rated at 2000 pounds capacity at $4^1/_3$% consistency. A view of one of these beaters is shown in Fig. 1. The tub size is 30 feet long by 11 feet 3 inches wide and the volume is 780 cubic feet. The tubs are constructed of stainless steel with tile bottoms. The beater roll is 72 inches in diameter, with a 72-inch face and weighs 2 tons. It is rotated at 140 r.p.m. This gives a peripheral speed of 2640 f.p.m. The beaters were installed with 300-hp. induction motors which operate at 585 r.p.m.

The beater roll is fitted with 192 $^1/_4$-inch bars. Space between the bars is about 1 inch. Each beater is fitted with three diagonal-type forged steel bedplates arranged over a 75° arc around the circumference of the roll. Each bedplate is $15^3/_4$ inches wide and has 25 bars. The plates with which the beaters were originally fitted had bars $^1/_4$-inch wide, spaced $^3/_8$ inch apart, placed at an angle of 45° with the roll bars. A portion of the experimental work was done with a different type bedplate having bars arranged in a herringbone pattern. In these plates, the same number of bars were used, but the bars were slightly narrower ($^3/_{16}$ inch) and arranged at a 25° angle with the roll bars.

Instrumentation of the beaters is quite complete. Adjustment of roll load to values higher or lower than actual roll weight is accomplished by means of pneumatic cylinders. Roll-load recorders are provided and any desired roll loading schedule may be obtained automatically by means of cam controllers. Handwheels are also provided for manual regulation of roll load. Stock supply and beater dump valves are operated by pneumatic cylinders. Each beater has two

Fig. 1. View of Jones-Bertrams beater

Table I. Tearing Strength and Tensile Strength vs. Beating Time at Different Roll Loads

Roll weight, %	Consistency, %	Tearing strength, %, at indicated beating time, min. 0	10	20	30	40	Tensile strength, %, at indicated beating time, min. 0	10	20	30	40
					No. 3 Beater						
80	3.66	414	383	343	323	295	36.0	48.7	53.8	59.1	68.9
100	3.76	405	364	330	308	276	36.2	49.4	57.4	64.2	69.3
125	3.78	391	335	296	261	236	35.4	50.4	60.2	66.9	72.1
					No. 1 Beater						
125	4.01	357	313	296	255	263	24.4	38.2	50.7	56.6	68.1
160	3.86	356	323	268	238	219	24.4	43.0	54.9	66.5	71.2
200	3.81	359	274	241	227	195	26.6	51.6	62.1	62.1	68.0

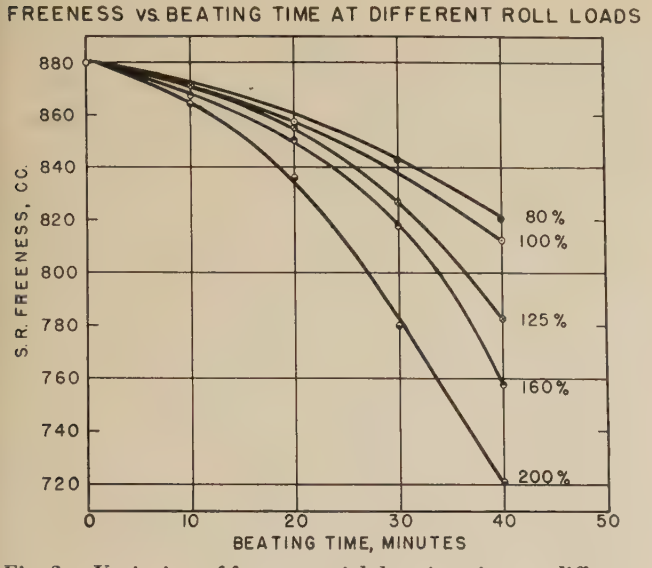

Fig. 2. Variation of freeness with beating time at different roll loads

dump valves so that it can be dumped into either of two stock chests. The load on beater motors is indicated by an ammeter at the instrument panel of each beater. In addition, a wattmeter for each beater is located in the power substation which serves the beaters. Recording wattmeters were attached whenever experimental runs were made.

The experimental study of the beaters was accomplished by means of special runs which were arranged during the course of regular production. The effect of the beating was determined by freeness measurements and by tests on handsheets. In such a study, the effects of day to day variations in stock of course could not be completely eliminated. However, steps were taken prior to the experimental runs to eliminate broke from the stock to the beaters. Also, periods of unusual mill conditions were avoided in scheduling runs.

At the beginning of each experimental run the beater was filled with slush pulp and the consistency was adjusted if necessary. The full roll load was applied immediately at the beginning of the cycle and maintained constant for a beating time of 40 minutes. Pulp samples were obtained before the roll was lowered and at 10 minute intervals thereafter. During the beating period ammeter readings were taken and the rate of circulation was determined. The temperature and pH of the stock were measured before and after the run. Roll load and power records were obtained.

Each pulp sample was tested for Schopper-Riegler freeness, and the permanganate number was determined on the initial sample from each run. Handsheets were made according to TAPPI Standard T 205 m-42, and these were tested for strength properties at standard conditions (73°F., 50% R.H.). The strength values are reported as percentage of the basis weight using the 24 × 36—480 ream.

DISCUSSION

The roll loads investigated were 80, 100, 125, 160, and 200% of roll weight. Average results were obtained on no less than three beater runs at each condition investigated. However, circumstances did not permit a thorough study of the entire roll load range in the same beater with the same tackle. Roll loads of 80, 100, and 125% were mainly studied in No. 3 beater

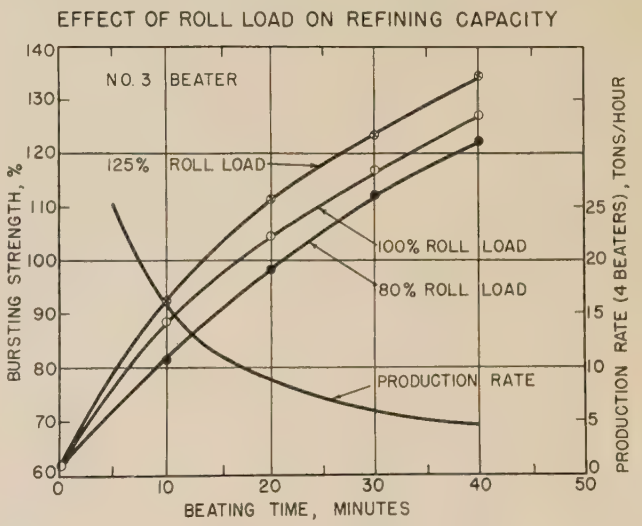

Fig. 3A. Effect of roll load on refining capacity in range of low roll loads

Table II. Tearing Strength and Tensile Strength vs. Power Consumption at Different Roll Loads

Roll weight, %	Tear strength, %, at indicated power consumption, kwh./ton pulp 0	40	80	120	160	200	Tensile strength, %, at indicated power consumption, kwh./ton pulp 0	40	80	120	160	200
					No. 3 Beater							
80	414	371	356	333	308	...	36.0	47.1	53.7	58.8	63.1	...
100	405	364	345	322	295	...	36.2	47.2	55.1	61.2	66.2	...
125	391	345	315	285	258	238	35.4	47.3	56.2	62.6	67.7	72.0
					No. 1 Beater							
125	357	332	306	283	263	248	24.4	35.1	44.0	51.8	59.1	66.7
160	356	326	396	268	246	229	24.4	37.1	46.2	54.2	61.2	68.7
200	359	318	283	251	224	212	26.6	43.0	52.9	58.2	62.0	65.0

Table III. Tearing Strength and Tensile Strength vs. Freeness at Different Roll Weights

Roll weight, %	Tearing strength, %, at indicated S.R. freeness, ml. 880	850	820	790	760	Tensile strength, %, at indicated S.R. freeness, ml. 880	850	820	790	760
				No. 1 Beater						
125	357	293	258	245	237	24.4	51.0	60.8	67.1	72.0
160	356	274	240	227	219	24.4	55.2	66.0	69.8	71.2
200	359	258	232	217	208	26.6	57.9	64.7	67.1	68.1

with the diagonal type bedplates, and loads of 125, 160, and 200% were studied in No. 1 beater with the herringbone type bedplates. In order to provide the necessary power to study 160 and 200% roll loads, the motor on No. 1 beater was rewound with glass insulated windings. A few runs at lower roll load were also made in No. 1 beater, but the results were adequate only to establish the relation between beating time and freeness.

Effect of Roll Load on Refining Capacity

In Fig. 2 curves are plotted showing the variation of freeness with beating time at the various roll loads in No. 1 beater. As would be expected, these curves show that the rate of freeness drop increases substantially with increased roll load. Thus, for example, at 80% roll load a freeness of 822 and at 200% roll load a freeness of 721 were obtained after a beating time of 40 minutes.

Although the development of strength with beating is related to the freeness drop, freeness and strength do not correlate under all conditions. Consequently, the direct variation of strength with beating time is of primary importance in evaluating beater refining capacity. In Figs. 3A and 3B the bursting strength at different roll loads and the production rate (for four beaters) are plotted against beating time. The production rate curve refers to actual average paper-machine production being derived from machine and beater operation records.

The bursting strength curves in Fig. 3A represent 80, 100, and 125% roll loads in No. 3 beater. The curves in Fig. 3B represent 125, 160, and 200% in No. 1 beater. The curves are shown separately because they were obtained in two beaters having different kinds of bedplates and thus are not exactly comparable. As the curves indicate, the bursting strength developed faster as roll load was increased. However, at roll

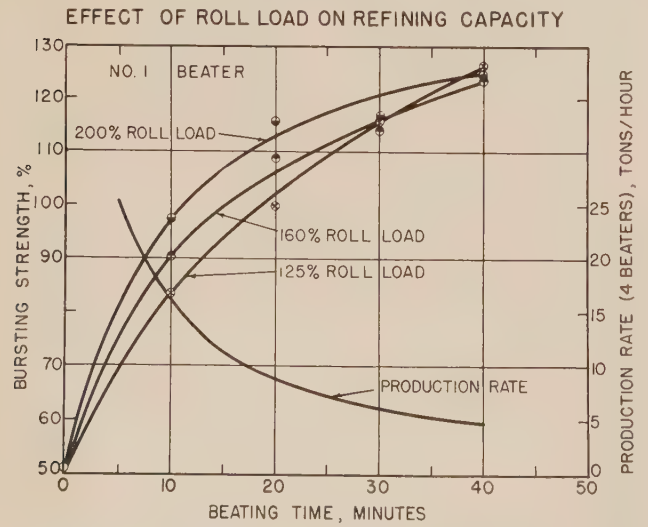

Fig. 3B. Effect of roll load on refining capacity in range of high roll loads

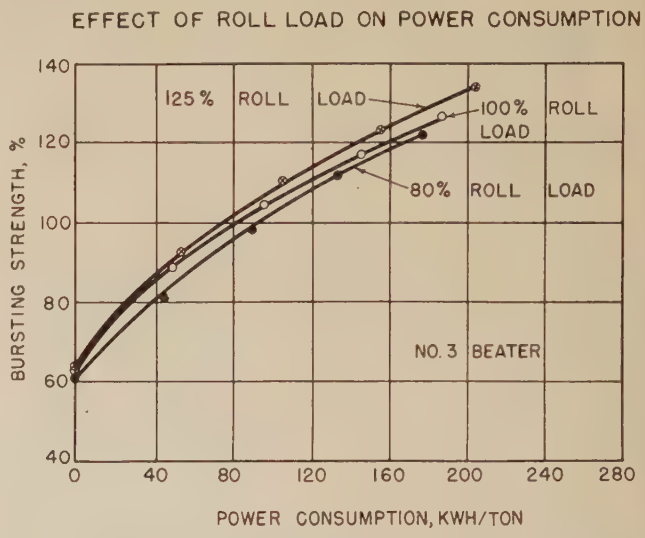

Fig. 4A. Effect of roll load on power consumption in range of low roll loads

loads over 125% the bursting strength increased with roll load only up to a certain beating time. For instance, the curve for 200% roll weight in Fig. 3B shows a rapid increase in bursting strength up to about 20 minutes beating time, after which the rate of development decreased until beyond 35 minutes beating time a lower bursting strength was obtained at 200% than at 125% roll load.

To show more directly the effect of roll load on refining capacity, one may refer to the beating times required to attain 100% bursting strength with the roll loads indicated in Fig. 3A. This degree of strength development would be attained in beating times equivalent to production rates of 8.5, 10.5, and 12.0 tons per hour for four beaters with roll loads of 80, 100, and 125%, respectively.

The influence of roll load on capacity may also be evaluated in terms of the increase in tensile strength and the decrease in tearing strength which usually occur in a manner which correlates with the development of bursting strength. In Table I are shown values for tensile and tearing strength which are based on the same beater tests as the bursting strength data represented in Figs. 3A and 3B. The average consistencies for the tests at each roll weight are also shown. The consistencies are considered to be sufficiently close to warrant comparison of the data. The data in Table I show that tensile strength increased and tearing strength decreased more rapidly as roll load was increased, except that in the case of tensile strength higher values were not obtained with 200% roll load after the longer beating times. This is similar to the result obtained for bursting strength as shown in Fig. 3B.

In general, it may be concluded that for beating times of less than about 25 minutes the refining capacity of the beaters may be increased by increasing the roll load to 200%. For longer beating times, no advantage is gained with roll loads greater than 125%.

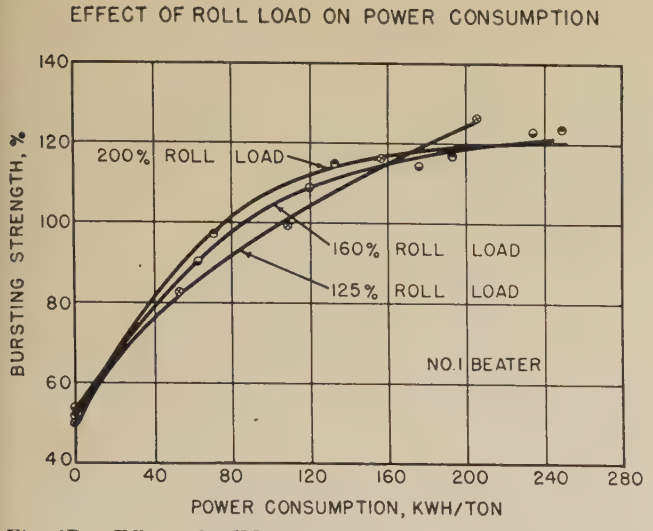

Fig. 4B. Effect of roll load on power consumption in range of high roll loads

Effect of Roll Load on Power Consumption

An increase of the roll load means an increased power input to the beaters. It is therefore of importance to take this factor into consideration and study the relationship between the power consumption and the roll load in regard to the strength properties.

In Figs. 4A and 4B, the bursting strength obtained at different roll loads has been plotted against the power consumption, kilowatt hours per ton of pulp. These curves show that to obtain a given strength less power is consumed at a higher roll load. Part of this power saving can be explained by the fact that during the beating the power required to rotate the roll and circulate the stock is constant and presumably independent of the roll pressure. Thus, if more load is applied, a greater part of the power will be consumed in actual beating. However, it is shown in Fig. 4B that when the beating was carried far enough, a point was reached where the curves for 160 and 200% roll load crossed the curve for 125%. This is also in line with the results of Fig. 3B which have been discussed. The fact that less power is consumed at higher roll loads is also confirmed by the changes in tear and tensile under the same circumstances. In Table II, average values for tear and tensile obtained at different power consumptions and roll loads are listed. From this table it can be seen that to obtain equal changes in tear and tensile values less power is consumed at higher roll loads. The lack of advantage for high roll loads at long beating times is again apparent in the case of tensile strength however.

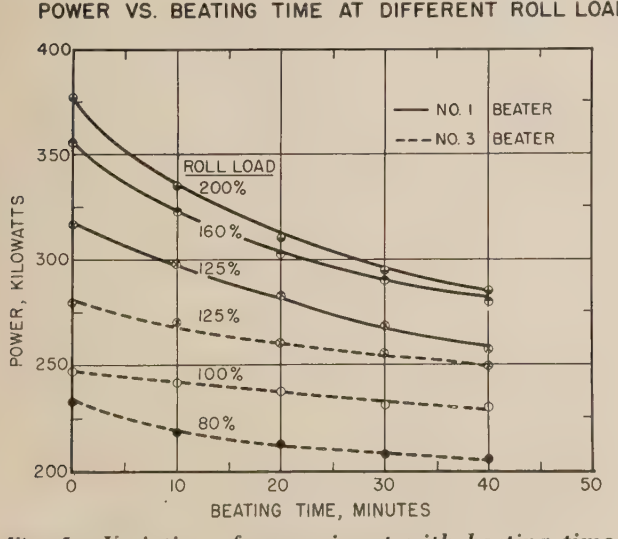

Fig. 5. Variation of power input with beating time at different roll loads

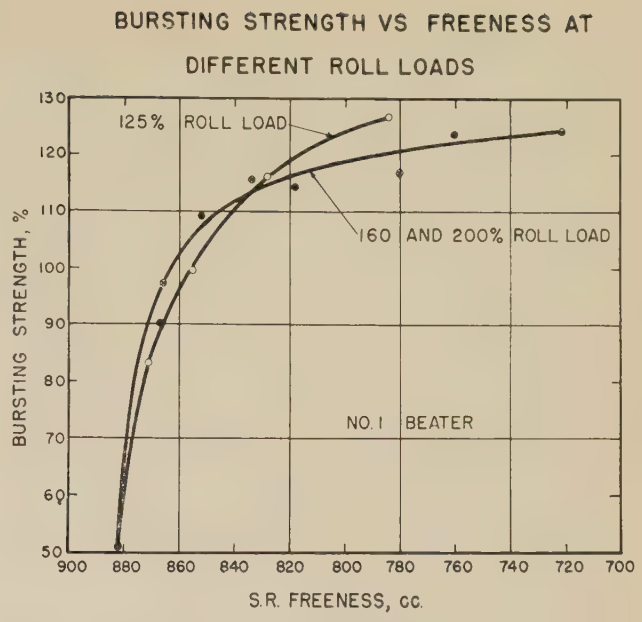
BURSTING STRENGTH VS FREENESS AT DIFFERENT ROLL LOADS
BURSTING STRENGTH, %
S.R. FREENESS, cc.
125% ROLL LOAD
160 AND 200% ROLL LOAD
NO. 1 BEATER

Fig. 6. Variation of bursting strength with freeness at different roll loads

In connection with the discussion of power consumption, Fig. 5 is of interest, as it shows the wattmeter readings at different beating times and roll loads. These curves and especially the one obtained at 200% roll weight show that the power input decreases rapidly during the first part of the beating cycle. At the end of 40 minutes the power input at 200% roll weight is nearly as low as that at 160%. Also it is to be noted that as roll load is increased above 125% of roll weight, the power increase is not proportional to the roll weight increase, but becomes successively less with each increment of added roll weight.

Effect of Roll Load on Quality

The consideration of increased roll load as a means of obtaining greater refining capacity must of course take

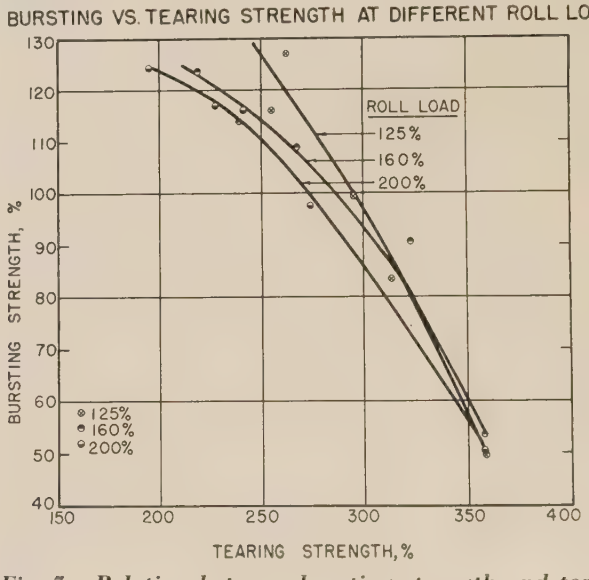

Fig. 7. Relation between bursting strength and tearing strength at different roll loads

into account the effect on the quality of the stock. In connection with this, the relation between strength and freeness is of importance. No significant differences were found in this relation for roll loads up to 125% nor between 160 and 200% roll load. However, some difference was apparent between 125% and the higher roll loads, as shown by the curves of Fig. 6. In the high freeness range, above about 830, higher bursting strength was obtained at a given freeness with the higher roll loads. In the lower freeness range this trend was reversed, that is, lower burst was obtained at a given freeness with higher roll loads. Comparable data showing the variation of tear and tensile with freeness at different roll loads are presented in Table III. The data for tensile strength show a reversal in the effect of roll load on the tensile-freeness relation, similar to the result shown for bursting strength in Fig. 6. However, tearing strength shows a tendency to be lower with increased roll load at a given freeness over the entire freeness range.

The relationship between burst and tear is probably the most important factor to be considered in connection with the effect of roll load on quality since it is desirable to minimize the sacrifice of tearing strength in the development of desired bursting and tensile strength values. Figure 7 shows this relationship between burst and tear in the 125 to 200% roll-load range. The points are quite scattered, but there is a definite tendency to lose tear as roll load is increased. The effect is greatest at higher degrees of strength development. For the roll-load range of 80 to 125% no difference could be found in the burst-tear relationship.

SUMMARY

In the beating of southern kraft pulp in Jones-Bertrams beaters it was found that the roll load could be increased to 125% with resulting increase in refining capacity and decrease in power consumption per ton of pulp and with no apparent limitation on beating time. For beating cycles less than about 30 minutes further increase in refining capacity and decrease in power consumption was obtained with roll loads up to 200%. However, some loss in tear at a given bursting strength was evident for roll loads above 125%. For beating times longer than about 30 minutes there appeared to be no advantage in roll loads greater than 125% with regard to either capacity, power consumption, or quality.

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The Preparation and Properties of Resin Saturating Papers

M. P. SEIDEL

The conversion of eight commercial pulps into phenolic resin saturation-type papers is described. Tests were made on the pulp and the paper. Machine conditions under which the papers were made are described.

One of the objectives of the laminating industry has been the formulation of a suitable set of paper-testing procedures which could be used to predict the performance of laminated materials. The literature on the correlation of paper and laminate properties is limited (*1*) and it is the objective of this report to present the formation methods employed and test values obtained for a number of resin saturation-type papers which are of interest to the laminating industry.

The papers which were studied were made from eight different wet pulp laps supplied by six different paper mills. Each of the laps was typical of the pulp used by the mill in making commercial paper runs. These pulps were then converted into paper by the Forest Products Laboratory on an experimental fourdrinier machine. Twenty papers were prepared from these pulps; the major variables which were investigated were the effects of jordan processing, thickness, and bulk. At this time only those papers which were prepared from the original pulps will be discussed.

Insofar as possible the papers were "square," that is the tensile strengths parallel and normal to the paper machine direction were identical. Minimum paper machine pressures, speeds and temperatures were used during paper formation, the objective of this phase of the project was the preparation of the most homogeneous paper possible from each of the pulps.

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PAPER FABRICATION

The wet laps were beaten only enough to impart sufficient strength to the finished paper to carry it through the resin-treating machine. Table I lists the pulps which were processed as well as the stock solids and freeness, the drier temperature, and the final thickness. An examination of the pulps listed indicates the broad range of this program—the kraft papers have good mechanical properties, the sulphite and alpha papers are used for good electrical properties, and the soda and newsprint are low-cost papers. The solids as determined on the headbox stock varied from 0.3 to 0.5% on an oven-dry basis and the freeness of all stocks with the exception of the newsprint was in the range of 840 to 890 ml. A wire speed of 50 f.p.m. and a shake frequency of 330 cycles per minute was standard for all of the papers. A drier temperature of 220°F. was used

Table I. Paper Machine Data

Paper	Solids, %	Freeness, ml.	Drier temperature, °F.	Pressure, p.s.i.g. wet 1st	press 2nd	3rd
Southern kraft	0.28	885	240	15	11	10
News broke	0.42	370	220	4	5	1
Unbleached scd. Mitsch.	0.48	870	240	6	2	1
Bleached soda	0.48	840	220	18	3	0
Regular alpha	0.34	858	220	9	2	0
Bleached sulphite	0.46	870	222	7	2	1
Northern kraft	0.36	858	222	8	2	1
Soft alpha	0.35	865	222	9	2	1

Table II. Paper Characteristics

Paper	Thickness, mils.	Bulk	Ream weight 25 × 40—500 lb.	Tensile, p.s.i.
Southern kraft	11.6	High	77.7	1084
News broke	10.6	High	80.4	2014
Unbleached scd. Mitsch.	9.8	High	106.4	1350
Bleached soda	11.1	High	89.4	906
Regular alpha	9.6	Low	97.8	1453
Bleached sulphite	10.3	High	111.0	907
Northern kraft	10.7	High	91.0	2924
Soft alpha	10.0	High	95.8	1045

on six of the papers, the kraft and Mitscherlich were dried at 240°F. The wet press pressures for the three presses which were used are reported in Table I. These pressures are low although the pressures used for the southern kraft were higher than for the other papers.

The paper characteristics of the eight papers are reported in Table II. Paper thickness varied from 9.6 to 11.6 mils, and paper bulk was high in all cases except for the regular alpha. The ream weight varied from 78 to 111 pounds and the tensile strength from 900 to 2900 p.s.i.

The paper properties which were measured are summarized in Table III. All of these properties other than the alcohol penetration varied over wide limits. The density of the papers was in the range of 0.37 to 0.60 and the air porosity was from 0 to 6 seconds for all the papers except the newsprint. The papers act quite differently with respect to water and alcohol penetration, considerable variation in the papers is observed in water penetration although all of the papers have identical alcohol penetration. The ash content of the papers varies from 0.18 to 0.98%.

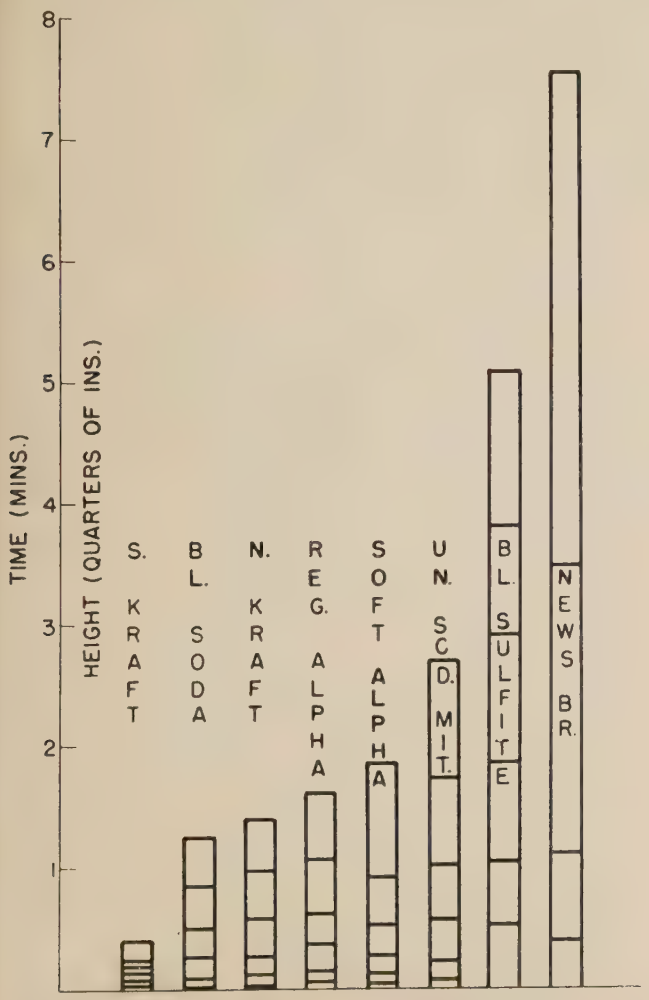

Fig. 1. Water capillary rise of laminating papers

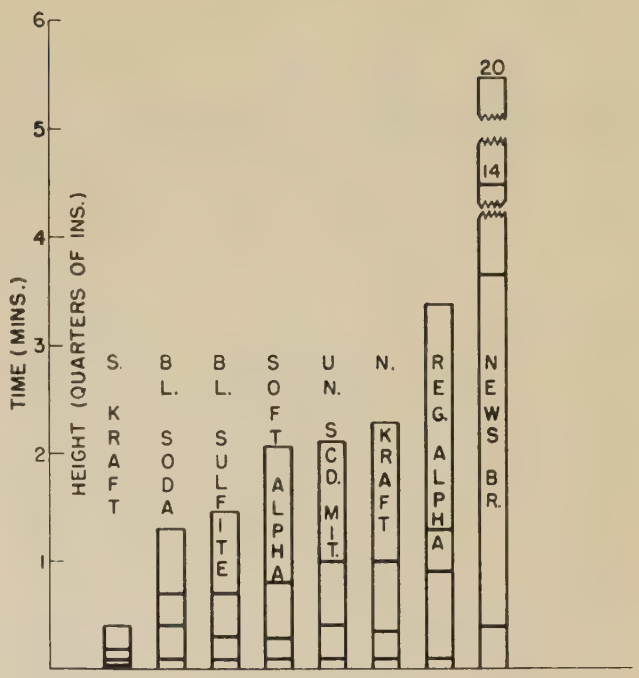

Fig. 2. Alcohol capillary rise of laminating papers

Table III. Paper Properties

Paper	Density, g./cc.	Porosity, air, sec.	Penetration, H_2O, sec.	Penetration, $EtOH_1$ sec.	Ash, av. %
Southern kraft	0.37	0	1	1	0.74
News broke	0.42	50	8	1	0.25
Unbleached scd. Mitsch.	0.60	6	3	1	0.55
Bleached soda	0.44	1.5	1	1	0.62
Regular alpha	0.56	3	1	1	0.20
Bleached sulphite	0.60	4	8	1	0.23
Northern kraft	0.47	4	3	1	0.98
Soft alpha	0.53	2	1	1	0.18

Figures 1 and 2 summarize the water and alcohol capillary rise of the papers. In these figures the time required for the liquid to wet the paper above the liquid surface in distances of fourths of an inch is summarized. No correlation between penetration and capillary rise is to be found for either water or alcohol. The capillarity determination tends to magnify differences in papers which are not evident in the penetration test.

CONCLUSIONS

A series of homogeneous laminating papers of known physical properties has been prepared. These papers have been thoroughly tested for homogeneity, porosity, and density, and attempts will be made to correlate these properties with certain properties of laminated boards made from these papers.

LITERATURE CITED

1. Barber, Robert W., *Tech. Assoc. Papers* 28: 297–300 (1945); *Paper Trade J.* 120, no. 19: 46–49 May 10, (1945).

The laminating papers described in this report as well as all of the test values were furnished by the Forest Products Laboratory at Madison, Wis. as part of a cooperative project with the Westinghouse Research Laboratories to study the fundamental properties of phenolic laminates.

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The Reaction Products of Lignin and Sodium Chlorite in Acid Solution

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In the digestion of wood with sodium chlorite and acetic acid, the lignin content of the original wood becomes almost completely soluble in the dilute acetic acid solution. Acidification with sulphuric acid precipitates a portion of the material and ether extraction of the filtrate gives other fractions. The precipitate has been separated into fractions, designated as chlorite lignin A and chlorite lignin B. The former has the formula $C_{26}H_{28}O_{14}Cl$, is free of polysaccharides, and appears to contain two methoxyl groups, two carboxyl groups, two hydroxyl groups, and a chlorine atom per unit weight of 600. The latter has the formula $C_{21}H_{21}O_{13}Cl_2$ and appears to contain one methoxyl group, two carboxyl groups, and two chlorine atoms per unit weight of 553. These two compounds seem to be chemical entities. The ether-soluble material contains fumaric, oxalic, and chloroacetic acids and a neutral crystalline wax. The reaction products from an isolated lignin (Indulin) differ somewhat from those obtained from wood, in that they contain fewer methoxyl groups and chlorine atoms.

Sodium chlorite has only recently become readily available. For that reason, general interest in and fundamental work on its behavior toward lignin and other organic compounds has necessarily been limited in scope.

In view of the mild action of acidic solutions of sodium chlorite on cellulose and its specific degradative action on lignin, the majority of the investigators (*1–9A*) had as their objective the preparation of a pulp or holocellulose more nearly representing the total carbohydrate fraction of the woody tissue. Others (*10–17A*) have found it useful as a bleaching agent for pulp where a strong product was desired.

Schmidt, et al. (*19–28*) have reported a large number of experiments on the preparation of the "Skelettsubstanz" (skeleton substance) from a number of woods and plants using aqueous solutions of chlorine dioxide. In one of these (*21*), the removed incrustants—i.e., lignin and some carbohydrate material—were separated into alcohol-soluble and alcohol-insoluble fractions, but nothing more was done with this material. Heuser and Merlau (*28A*) carried out similar experiments to prepare the "Skelettsubstanz." They dialyzed the aqueous solutions of incrustants, evaporated the residual liquor to dryness, and analyzed the residues (25% yield based on the wood) for pentosans, obtaining around 8%.

Fuchs and Honsig (*29*) treated several isolated lignins (including Willstätter lignin) with chlorine dioxide solutions, obtaining water-soluble products and a residue which they also separated into alcohol-soluble and alcohol-insoluble fractions.

Sarkar (*30*) treated jute lignin with aqueous chlorine dioxide and claimed the chlorinated product was identical with that obtained by the use of chlorine.

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Sohn and Reiff (*1*) tried acidifying with strong acids the clear liquors obtained when wood was digested with acidic solutions of sodium chlorite. They obtained a white flocculent precipitate which they believed to consist of hemicelluloses. The decomposition of the lignin complex was thought to have been very thorough and the recovery and utilization of this constituent impossible.

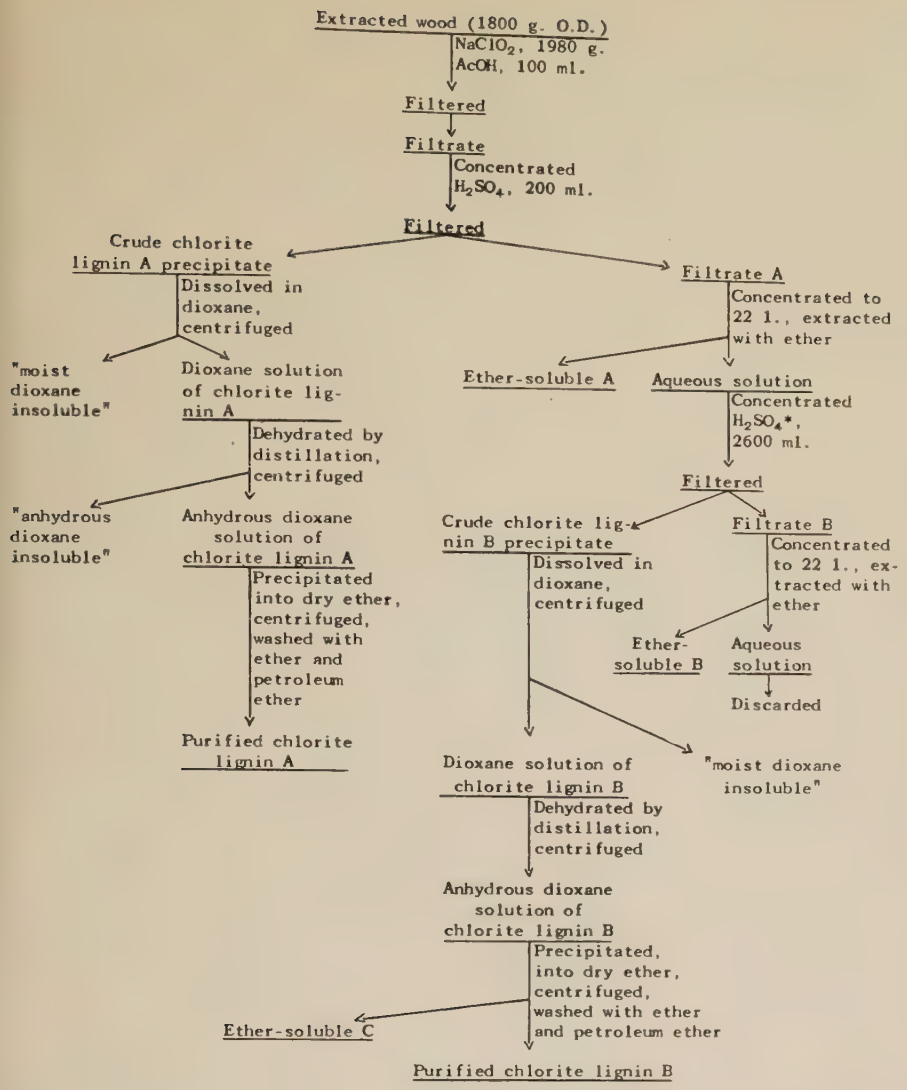
The observations of Sohn and Reiff were followed by a more extensive investigation by Jayme and co-workers (*5, 31, 32*). In a preliminary paper Jayme, Eser, and Hanke (*31*) expressed the belief that the white precipitate obtained on acidification of chlorite liquor was not a pure polysaccharide but contained a lignin residue. Jayme and Hanke (*32*) extended this work and published a comprehensive paper on the properties of the nonholocellulose portion of the wood-chlorite reaction mixture. These authors purified their residues by dialysis and obtained products having lignin characteristics, but which yielded an appreciable amount of glucose upon hydrolysis with acid. On the basis of this work, Jayme and Hanke suggested the presence of an "excess polysaccharide" in wood and postulated a structure for a ligno-carbohydrate complex which explained the origin of this material, the formation of acid lignins, the formation of phenylpropane compounds, and lignin reactions. They concluded that native spruce lignin is a "dehydrated polysaccharide of the hexose-type, substituted preponderantly with guaiacyl residues."

The most recent work in this field was done by Pearl (*33*) and Pearl and Wise (*34*) who isolated 6-chlorovanillin from the steam nonvolatile aldehyde fraction of the ether extract of the waste liquor from the digestion of black spruce with sodium chlorite and acetic acid. Acidification of this liquor with strong mineral acids prior to ether extraction precipitated the white flocculent material described by Jayme and Hanke (*32*) and by Sohn and Reiff (*1*).

EXPERIMENTAL

The wood chosen for this study was slash pine (*Pinus caribaea* Morelet) because of its low extractive content, ready availability, and the fact that it had already been used to some extent in chlorite holocellulose studies. The wood was received in the form of freshly cut sticks 8 inches in diameter; it was 15 to 17 years old and contained about 5% heartwood. The logs were barked and chipped, and the air-dried chips were reduced in a hammer mill and then in a Wiley mill. The resulting wood meal was screened and the 10 to 40-mesh fraction collected.

The wood was pre-extracted (15 days) with a 1:3 95% ethanol-benzene mixture in a large continuous extractor assembly constructed for this purpose. The


Extracted wood (1800 g. O.D.)
$NaClO_2$, 1980 g.
AcOH, 100 ml.
Filtered
Filtrate
Concentrated H_2SO_4, 200 ml.
Filtered
Crude chlorite lignin A precipitate
Dissolved in dioxane, centrifuged
"moist dioxane insoluble"
Dioxane solution of chlorite lignin A
Dehydrated by distillation, centrifuged
"anhydrous dioxane insoluble"
Anhydrous dioxane solution of chlorite lignin A
Precipitated into dry ether, centrifuged, washed with ether and petroleum ether
Purified chlorite lignin A
Filtrate A
Concentrated to 22 l., extracted with ether
Ether-soluble A
Aqueous solution
Concentrated H_2SO_4*, 2600 ml.
Filtered
Crude chlorite lignin B precipitate
Dissolved in dioxane, centrifuged
Filtrate B
Concentrated to 22 l., extracted with ether
Ether-soluble B
Aqueous solution
Discarded
"moist dioxane insoluble"
Dioxane solution of chlorite lignin B
Dehydrated by distillation, centrifuged
Anhydrous dioxane solution of chlorite lignin B
Precipitated, into dry ether, centrifuged, washed with ether and petroleum ether
Ether-soluble C
Purified chlorite lignin B

* Before the second acidification, the liquor was blown with steam to remove residual ether, and the concentrated acid was diluted with water to decrease the heat of solution when added to the liquor.

Fig. 1. Flow sheet, cook 8

extracted wood contained 0.33% extractives, 27.65% lignin, and 0.14% ash.

In orienting experiments it was found that the precipitate obtained by acidification of the chlorite liquor could be purified by dissolving in dioxane and precipitating the dried dioxane solution into dry ether. Two products were obtained (termed "chlorite lignins A and B") in a yield of about 5% (on the basis of the original lignin in the wood). Ether-soluble materials were obtained by extraction of the liquor after the chlorite lignins were removed in a yield of 26% (based on the lignin in the wood).

After preliminary experiments, the final cooking conditions were as follows: About 28 liters of distilled water in a 10-gal. Pyrex battery jar were heated by direct steam to 72°C. The total volume was then about 30 liters. Eighteen hundred grams of wood meal (oven-dry basis) were added, giving a mixture of 6% consistency. This was followed by 25 ml. of glacial acetic acid, and then the first addition of 495 grams of sodium chlorite (the analytical grade supplied by The Mathieson Chemical Corp.). At hourly intervals thereafter, three more additions of 25 ml. of acetic acid and 495 grams of sodium chlorite were made; the total sodium chlorite addition was 1980 grams. The heat of reaction increased and maintained the temperature at 80°C. for the first 3 hours after which the temperature dropped slowly to 75°C. at 4.5 hours, the conclusion of the cook. Table I presents a typical cooking schedule (for cook 8), and Table II gives the experimental data and yields for all cooks.

After 4.5 hours, the hot mixture was filtered on two 250-mm. Buchner funnels, an operation which required about 30 minutes. The filtrate was aerated overnight to remove excess chlorine dioxide and the clear liquor was acidified by the addition of 200 ml. of concentrated sulphuric acid. The flocculent precipitate which formed was allowed to settle and the supernatent liquor was decanted by siphoning. The precipitate was filtered on a 90-mm. medium porosity fritted glass funnel. Figure 1 is a flow sheet which illustrates this and subsequent operations. The exact details of the separations are omitted from this discussion.

Investigation of Dioxane-Insoluble Material

It was suspected that the dioxane-insoluble material would be predominantly carbohydrate, so a typical sample was subjected to acid hydrolysis. A 6.5-gram sample of the "moist dioxane-insoluble" fraction from cook 7 chlorite lignin A was treated with 100 ml. of 5% sulphuric acid. The mixture was refluxed for 3 hours, filtered, and the residue washed acid free. The hydrolyzate was neutralized with barium carbonate and filtered. The filtrate gave a strong test with Fehling solution. Munson-Walker sugar determinations showed that 1.35 grams of reducing sugars (as glucose) were present in the fraction, or only 0.07% based on the weight of the wood.

Mannose was identified in the hydrolyzate as its phenylhydrazone; 0.35 gram of mannose was present in the fraction. Galactose was also identified by the formation of mucic acid; 0.22 gram of galactose was present. A positive test for uronic acids was also obtained with napthoresorcinol. The hydrolysis residue amounted to 0.7 gram and contained 3.0% ash and 8.63% methoxyl, indicating that it was principally ligninlike material. The presence of this small quantity of carbohydrate in the chlorite lignin A precipitate was probably due to physical occlusion. It could not have been involved in a chemical union with the chlorite lignin A since it was separated by the action of an inert solvent.

Analysis and Investigation of Chlorite Lignins

To obtain a sufficient quantity of a uniform chlorite

Table I. Typical Cooking Schedule

Time, hr.	Acetic acid added, ml.	Sodium chlorite added, g.	pH	Temperature, °C.
0	25	495	..	72
0.75	..	..	4.20	..
1.00	25	495	..	..
1.25	..	..	4.50	79
1.75	..	..	4.35	80
2.00	25	495	..	..
2.25	..	..	4.45	80
2.75	..	..	4.50	80
3.00	25	495	..	..
3.25	..	..	4.55	79
3.75	..	..	4.75	77
4.00	..	..	..	75
4.50	Commenced filtering		4.70	..

Note: Eighteen hundred grams of wood (oven-dry basis) were used.

lignin A material for future work, the products of cooks 8 and 9 were combined and purified to constant methoxyl content. The final purified material is a white, amorphous powder containing 10.5% methoxyl, 5.95% chlorine, and 0.3% ash. On the melting point block, chlorite lignin A softens at 165 to 170°C., darkens in color at 175 to 180°C., and finally changes to a clear, brown, tacky resin at 200°C.

The yields of chlorite lignin B from cooks 1, 7, and 8 were combined and purified to constant methoxyl content. The final product was an amorphous powder with a light tan color and contained 5.6% methoxyl, 13.2% chlorine, and no ash.

Both chlorite lignin products exhibit similar solubility behavior, being soluble in acetone, methanol, butanol, pyridine, dioxane, dilute alkali, sodium bicarbonate solution (with the liberation of carbon dioxide) and insoluble in benzene, ether, and petroleum ether.

Ultraviolet Absorption Spectra

Three milligram samples of chlorite lignins A and B were each dissolved in 100 ml. of absolute methanol, and ultraviolet absorption data were obtained with a Beckman Model DU spectrophotometer set at minimum slit width. The resulting data are presented in Fig. 2, together with a curve for isolated spruce native lignin (using dioxane as a solvent) for comparison. No pronounced inflections are noticeable in the curves for either chlorite lignin A or B, in contrast to those of native lignin and many other lignin preparations.

Potentiometric Titration Data

The pronounced acid nature of the chlorite lignin products, as indicated by the liberation of carbon dioxide from a sodium bicarbonate solution, prompted an attempt to determine the neutralization equivalent of these materials by potentiometric titration in alcohol-water solution with sodium hydroxide. Although the titration curve for chlorite lignin A (Fig. 3) showed a noticeable break, the curve for chlorite lignin B rose steadily until a pH of 11 was approached asymptotically. Taking the end point at pH 9.5, the neutralization equivalents can be calculated as 410 and 345 for chlorite lignins A and B, respectively. Because of the uncertainty involved in the location of a proper end point, these values are only approximate.

Hydrolysis Experiment on Chlorite Lignin A

If chlorite lignin is or contains a ligno-carbohydrate complex, it should be possible to split off the polysaccharide portion by acid hydrolysis. A 2-gram sample of chlorite lignin A was refluxed for 6 hours with 100 ml. of 5% sulphuric acid. Most of the lignin was recovered as a brown, brittle resin (yield, 1.85 grams). The hydrolyzate was neutralized with barium carbonate, filtered, and tested with Fehling solution with negative results.

As a more vigorous hydrolytic treatment, a 1-gram sample of chlorite lignin A was treated with 20 ml. of cold 72% sulphuric acid and kept at 18 to 22°C. for 2 hours (as in the Klason lignin determination). The material dissolved readily giving a dark red-colored

Table II. Summary of Experimental Conditions and Yields

Cook number	1	2	3	4	5	6	7	8	9
Total glacial acetic acid used, ml.	360	360	360	360	360	360	90	100	100
Final liquor pH	3.7[a]	[b]	[b]	[b]	3.8	3.8	4.65	4.70	4.70
Amount concentrated sulphuric acid used, first acidification, ml.	300	100	100	3600[c]		None	200	200	200
Amount of concentration	to 22 l	to 9 l[d]	to 9 l[d]	to 22 l	[e]	to 4 l[f]	to 22 l	to 22 l	to 22 l
Yield, ether-soluble A, g.	63.0	46.0	35.5				33.0	37.0	43.3
Yield, ether-soluble B, g.	126.8	113.0		204.6[c]			141.5	132.2	178.0
Yield, ether-soluble C, g.	8.0	28.0		28.1			13.6	6.9	8.1
Amount concentrated sulphuric acid used, second acidification, ml.	2200	2200					3000	2600	2600
Yield, chlorite lignin A, g.	26.3	20.0	12.5				30.3	49.5	43.8
Yield, chlorite lignin B, g.	6.1	12.8		16.7[c]			10.5	3.1	2.0
Yield, "moist dioxane insoluble" from chlorite lignin A, g.	3.8	5.6	5.0				6.9	7.3	10.9
Yield, "anhydride dioxane insoluble" from chlorite lignin A, g.	3.4	5.5	3.1				7.2	11.7	11.5
Yield, "moist dioxane insoluble" from chlorite lignin B, g.	5.3	6.2					5.6	3.3	3.1
Yield, holocellulose, %[g]	71.7	73.7	71.7	67.8	69.2	68.7	72.9	73.2	72.7
Ash in holocellulose, %	1.03	1.52	1.45	1.02	0.65	0.72	1.07	1.14	1.11
Lignin in holocellulose, %	1.54	1.89	2.00	1.34		1.03	2.16	2.44	2.57
Ash-free yield holocellulose, %	71.0	72.6	70.6	67.2	68.7	68.2	72.2	72.4	72.0
Ash- and lignin-free yield holocellulose, %	69.9	71.2	69.2	66.9		67.5	70.7	70.5	70.2

Note: Ash on original wood was 0.14% and lignin content was 27.65%. Eighteen hundred grams (oven-dry basis) of wood were used in each cook.

[a] After 1.5 hours.
[b] Not measured.
[c] This was a one step acidification (enough acid added to promote secondary oxidation reaction) so that chlorite lignins A and B are combined into one product, as were ether solubles A and B.
[d] These liquors were neutralized before concentration, to see if the ether-soluble yield could be increased, but with no noticeable effect.
[e] Cook 5 liquor was not used.
[f] This liquor was prepared for a nitrobenzene oxidation, but was not used.
[g] Ash and lignin containing basis.

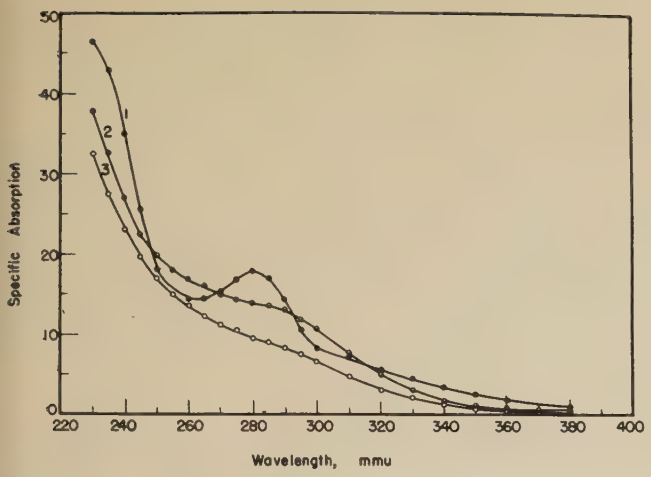


Fig. 2

solution; when poured into 750 ml. of distilled water, a creamy flocculent precipitate was obtained, which turned to a reddish brown during the subsequent 4-hour heating period at 70 to 75°C. The yield of lignin residue was 31.2% and the ash-free material contained 9.68% methoxyl and 6.50% chlorine. A sample of the neutralized filtrate from this experiment tested negative with Fehling solution.

Acetylation of Chlorite Lignins A and B

Two-gram samples of chlorite lignins A and B were each dissolved in 30 ml. of dry pyridine and 24 ml. of acetic anhydride were slowly added with shaking. After 48 hours at room temperature, the reaction products were precipitated by pouring the solutions onto cracked ice. The crude acetylated chlorite lignin A was dissolved in ten times its weight of dioxane and precipitated (5 ml. at a time) into 250-ml. portions of dry ether. This product was acetylated twice more in a similar manner. The crude acetylated chlorite lignin B was insoluble in dioxane but partially soluble in a 1:1 dioxane-acetone mixture. Accordingly, the crude material was dissolved in the mixed solvent and precipitated (10 ml. at a time) into 250-ml. portions of dry ether.

Acetylated chlorite lignin A contained 9.13% methoxyl, 14.5% acetyl, and 4.88% chlorine; acetylated chlorite lignin B analyzed 5.64% methoxyl, 15.4% acetyl, and 8.96% chlorine.

Methylation of Chlorite Lignins A and B with Diazomethane

Two-gram samples of chlorite lignins A and B in dioxane solution were methylated with diazomethane prepared from nitrosomethylurea. The methylations were repeated until the methoxyl content was reasonably constant. The methylated products appeared to be slightly lighter in color than the original materials, and were no longer soluble in cold alkali. Methylated chlorite lignin A contained 19.14% methoxyl and 4.56% chlorine; methylated chlorite lignin B contained 16.13% methoxyl and 9.17% chlorine.

In order to determine if phenolic or carboxylic groups, or both, are involved in the diazomethane methylation, a 0.5-gram sample of methylated chlorite lignin A was saponified by refluxing with 50 ml. of chloride-free 10% aqueous potassium hydroxide for one hour. A few milliliters were distilled from the mixture under reduced pressure to effect the removal of any methanol. About 0.5 ml. of the clear solution was acidified with 20% nitric acid, the precipitate filtered, and the clear filtrate tested for the presence of chloride with silver nitrate solution with negative results. Thus, the chlorine group in chlorite lignin A is stable to alkali and is present in a ring. The balance of the saponification mixture was then acidified with 10% sulphuric acid and the resulting precipitate was centrifuged, dried, dissolved in dioxane, and precipitated into dry ether in the usual way. Analysis of the saponified product showed that the methoxyl content had been decreased from 19.14 to 12.08%. This is an indication that the acid character of chlorite lignin A is due primarily to carboxyl groups.

Treatment of Chlorite Lignin A with Methanol and Hydrogen Chloride

If carboxyl groups are present, esterification with methanol and hydrogen chloride should be possible. A 2-gram sample of chlorite lignin A was dissolved in 40 ml. of absolute methanol and the solution saturated with anhydrous hydrogen chloride, followed by refluxing for 1 hour on a water bath. After standing overnight, the reaction mixture was precipitated into distilled water. The dried product was purified by dissolving 1.5 grams in 15 ml. of dry dioxane, and precipitating (5 ml. at a time) into 250-ml. portions of dry ether. The material contained 19.28% methoxyl and 7.68% chlorine. This high chlorine value is probably due to some chlorination by the hydrogen chloride. When corrected for this increase in chlorine content, the methoxyl value is 19.7%. These data suggest that either carboxyl groups are present in the chlorite lignin A unit or that acetal formation has taken place involving a carbonyl group.

If the reaction just described is an esterification, it should be possible to recover the original lignin product by saponification. Accordingly, a 0.7-gram sample of this derivative was refluxed 1 hour with 50 ml. of

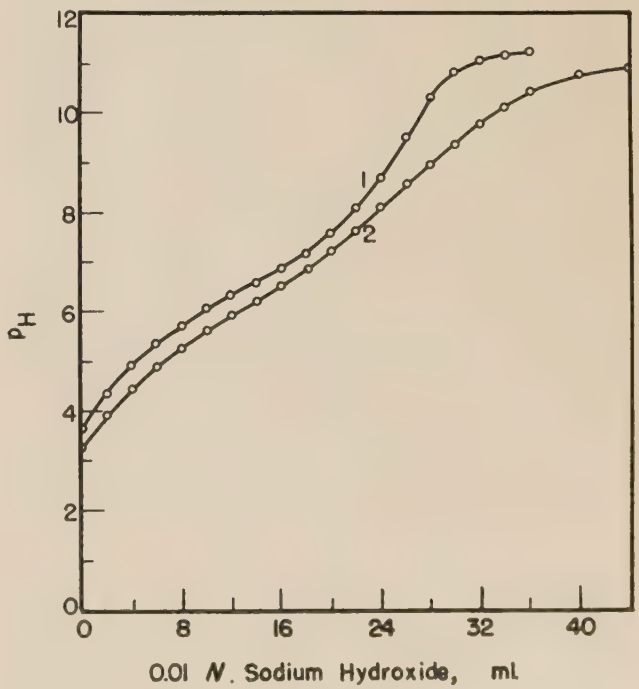


Fig. 3

aqueous 5% potassium hydroxide and allowed to stand overnight at room temperature. The saponification mixture was subjected to vacuum distillation to remove any methanol that may have formed and acidified. The resulting precipitate was handled in the same manner as in the saponification of the methylated chlorite lignin A. The purified product contained 10.71% methoxyl and 5.45% chlorine. Since the methoxyl and chlorine contents of the original chlorite lignin A is 10.5% and 5.95%, respectively, it is evident that only esterification took place since the methyl groups added have been quantitatively removed by saponification. It may be noted also that the chlorine introduced during esterification by the action of the hydrogen chloride was removed.

INVESTIGATION OF THE ETHER-SOLUBLE MATERIALS FROM THE CHLORITE LIQUORS

Fractionation of Ether-Soluble A Fractions

The yields of the ether-soluble A fractions from the various cooks are shown in Table II. When freshly prepared, these materials were a tarry, partially crystalline mixture of a light-orange color. On standing, the substances darkened in color and became partly insoluble in ether. The fractionation of a representative batch of the material from one cook (cook 1, 63.0 grams) was carried out by separating into 21% sodium bisulphite-soluble, 8% sodium bicarbonate-soluble, and 5% sodium hydroxide-soluble products (plus the remainder, or neutral fraction) according to conventional methods.

The bisulphite-soluble fraction amounted to 9.5 grams. On standing, colorless needles separated from this residue. Upon recrystallization from methanol-chloroform, they melted at 100°C. An aqueous solution of the compound was acidic. This suggested oxalic acid, and its identity was confirmed by a mixed melting point with an authentic sample of oxalic acid dihydrate.

The bicarbonate-soluble fraction was obtained as a partially crystalline, partly tarry mixture amounting to 7.8 grams. The crystalline material was filtered and recrystallized from methanol-chloroform. This substance behaved in the same manner as the fumaric acid obtained by filtration on the original ether-soluble A fractions of cooks 7, 8, and 9, the identification of which will be described later. The alkali-soluble fraction was a dark brown, pungent smelling tar (1.8 grams) which was not investigated further.

The neutral residue was leached with a little hot ethanol. On cooling, a white crystalline material separated. After several recrystallizations from absolute ethanol, a white, waxy crystalline compound was obtained which melted at 57 to 58°C. The elementary analysis gave C 81.74% and H 14.32%, which suggests the formula $C_{26}H_{53}OH$(C 81.70%, H 14.13%).

Although the melting point checks for stearyl alcohol ($C_{18}H_{37}OH$), the analytical data suggest a longer chain. In all probability, this is a mixture of an alcohol and a long chain hydrocarbon. The origin of such a material may be the original extractives of the wood.

Identification of Fumaric Acid

The fine, granular crystalline material obtained in the bicarbonate-soluble fraction of the ether-soluble A fraction from wood and the bisulphite-soluble fraction of ether-soluble A from Indulin (to be described later) appeared to be the same substance that had settled out of the original ether-soluble A residue. The ether-soluble A fractions of cooks 7, 8, and 9 were filtered and the crude material was recrystallized from a 1:5 methanol-chloroform mixture. A sample of this material (from cook 7) was recrystallized twice, and yielded fine, fluffy crystals melting at 287°C. (sealed capillary). On a melting point block, the compound started to sublime around 205°C. Its neutralization equivalent, determined by titration with sodium hydroxide to the phenolphthalein end point, was 55. This information, together with ultimate analysis (C 41.51%, H 3.61%), suggested fumaric acid.

Isolation of Monochloroacetic Acid

When most of the ether had been removed in concentrating the ether-soluble A fraction, a higher boiling liquid distilled which was apparently a mixture of acetic acid and a chlorinated acetic acid. The highest boiling fraction solidified on cooling and formed large, colorless prisms which melted sharply at 50°C. The reported melting point of the gamma form of monochloroacetic acid is 50.05°C. A mixed melting point with an authentic sample of monochloroacetic acid showed no depression. This compound may originate from the chlorination of the acetic acid used to acidify the wood reaction mixture, although an attempt to form it directly by the action of sodium chlorite on acetic acid failed. Another possibility is the chlorination of side chains from lignin and subsequent oxidation.

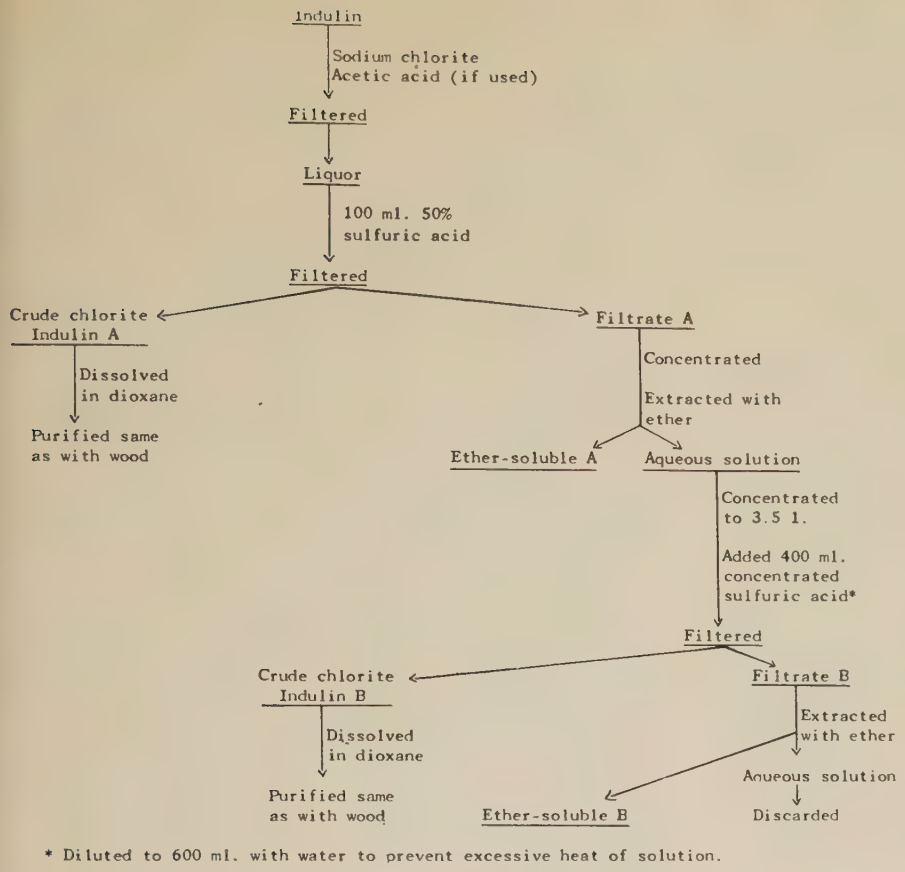
Isolation of Oxalic Acid Dihydrate from the Ether-Soluble B Fraction

After standing for a few weeks, colorless needles began to separate from the viscous brown tars of the ether-soluble B fractions. One of these (cook 7) was filtered to separate the crystalline product from the residual tar. This compound was recrystallized from methanol-chloroform. From its neutralization equivalent (61), its melting point (99 to 100°), and its elementary analysis (C 19.22%, H 4.72%), this is oxalic acid dihydrate.

It is not known whether oxalic acid is derived from the oxidation of the small amount of carbohydrates solubilized by the chlorite liquor, from the lignin, or from both.

SODIUM CHLORITE-INDULIN REACTIONS

The purpose of these experiments was to determine whether the action of sodium chlorite on an isolated lignin would give rise to products similar to those obtained from the chlorite digestion of wood. The southern pine kraft lignin product "Indulin A" of the West Virginia Pulp and Paper Co. was used for this purpose. The original Indulin contained 13.90% methoxyl, 1.40% sulphur, and 0.22% ash. The following method was adopted for the Indulin reactions with sodium chlorite. Two liters of distilled water (with or without the addition of acetic acid) in a 4-liter Erlenmeyer flask were heated to approximately 70°C. Fifteen grams of sodium chlorite were added, followed by the slow addition of 5 grams of Indulin. In about 15 minutes the reaction subsided, the Indulin having gone completely into solution. The heat of reaction kept the temperature of the mixture at 75 to 85°C.



* Diluted to 600 ml. with water to prevent excessive heat of solution.

Fig. 4. Flow sheet for Indulin reactions

Two similar additions of sodium chlorite and Indulin followed at about 15-minute intervals. Fifteen grams of Indulin were thus oxidized in each run, and the runs were repeated until 90 grams of Indulin had been consumed for each experiment. In experiment 1, a total of 3 ml. of glacial acetic acid were used to acidify the reaction mixture from 90 grams of Indulin. Experiment 2 was a duplicate run, except that no acetic acid was used. In experiment 3, 4 parts of sodium chlorite were used to 1 part of Indulin in the absence of acetic acid. This was done to determine the effect of an excess of sodium chlorite since it had been previously observed that the least amount of sodium chlorite necessary to solubilize the Indulin was about three times the weight of the lignin product. Table II summarizes the experimental conditions and the yields of various fractions.

The liquors from these reactions were acidified and the products handled in a manner analogous to those from wood. Figure 4 is a flow sheet illustrating the separations.

Analysis of Chlorite-Indulin Products

The chlorite Indulin A and B products (corresponding to chlorite lignins A and B) were purified and analyzed. Table III summarizes the methoxyl and chlorine data after the last purification step.

Table III. Analysis of Chlorite-Indulin Products

Experiment	*Chlorite-Indulin A*		*Chlorite-Indulin B*	
	Methoxyl, %	*Chlorine, %*	*Methoxyl, %*	*Chlorine, %*
1	8.56	3.77	3.90	9.26
2	8.11	3.76	3.40	9.55
3	8.23	4.77	2.20	12.13

The presence or absence of acetic acid used in the reactions appeared to have little effect on the methoxyl and chlorine content. Increasing the sodium chlorite to Indulin ratio increased the amount of chlorination with little effect on the methoxyl value but decreased the yield markedly. On comparing these products with the corresponding ones prepared from wood, it can be seen that the chlorine and methoxyl contents of the Indulin products were somewhat lower than in the case of chlorite lignins A and B. This might be expected, inasmuch as the Indulin lignin had undergone two degradative processes during its isolation.

Ultraviolet absorption spectra of these products exhibited the same features as those described for the chlorite lignin products.

Fractionation of Ether-Soluble A from Indulin-Chlorite Liquors

The three ether-soluble A fractions from the three Indulin experiments were combined (totaling 51.3 grams), dissolved in ether, and separated into four fractions as with the wood products. The bisulphite-soluble fraction (14.7 grams) was obtained as a tarry, partially crystalline residue. The crystalline matter was filtered and recrystallized from methanol-chloroform. The product exhibited the same appearance, sublimation behavior, and melting point (287 °C. in a sealed capillary) as fumaric acid. A melting point with an authentic sample of fumaric acid showed no depression.

The bicarbonate-soluble fraction (6.2 grams) was also filtered and the crystalline material was recrystallized from methanol-chloroform. A white, powdery product was obtained which did not melt up to 300 °C. in a sealed capillary. The small yield of pure material obtained prevented further investigation.

The alkali-soluble fraction (1.5 grams) was a dark brown tar of pungent odor and was not investigated further. The neutral fraction (2.5 grams) was an oily, tarry mixture which yielded a white, waxy, crystalline material when leached with hot ethanol. This substance melted at 57 to 58 °C. and is evidently the same long chain alcohol or mixture of compounds as obtained from the neutral fraction from wood.

DISCUSSION OF EXPERIMENTAL DATA

Chlorite lignin A contains 5.95% chlorine and 10.5% methoxyl. Therefore, approximately one chlorine atom is present for every two methoxyl groups in the smallest molecular building unit. If the molecular weights of these groups are divided in turn by their analytical percentages, values of 597 and 590 are obtained based on chlorine and methoxyl, respectively. These figures are called "unit weights," since no evidence has been presented that they are true molecular weights.

When the methyl ester of chlorite lignin A was formed, the methoxyl content was approximately doubled. Therefore, we can assume that two methyl groups had been introduced. A unit weight may be calculated from these data by use of the following formula:

$$P = [(31n + 31m)/(M + 14m)]100,$$

where

n = Number of methoxyl groups originally present
m = Number of methyl groups introduced
M = Unit weight
P = Analytical percentage methoxyl of the derivative

When solved for M with P equal to 19.7 and n and m equal to 2, the value obtained is 602.

In the same manner, assuming two methyl groups were introduced in the diazomethane methylation to give a final methoxyl content of 19.2%, the formula yields the figure 617.

When chlorite lignin A was acetylated, the methoxyl content decreased from 10.5 to 9.13%. Assuming this decrease to be due only to an increase in the molecular weight, it can be calculated from the latter value that the unit weight had been increased to 680. The molecular weight of two acetyl groups introduced is 84; subtracting this from 680 gives a value of 594 for the unit weight of the product before acetylation.

The average of the five values for a unit weight given above is 600. The good agreement obtained with the individual values indicates that chlorite lignin A may be a chemical entity; therefore, it is of interest to calculate a probable empirical formula. The formula $C_{26}H_{28}O_{14}Cl$ has a molecular weight of 599.5 and the experimental and calculated values for elemental composition are compared in Table IV.

The above experimental data indicate that certain functional groups are present in the chlorite lignin A structure. On the basis of a unit weight of 600, the presence of one chlorine and two methoxyl groups was established by analysis. The esterification experiment, followed by saponification, indicates that two carboxyl groups are present. The acetylation experiment showed the presence of two hydroxyl groups since two acetyl groups were introduced. These could have been either aliphatic or phenolic hydroxyl groups. If phenolic hydroxyl groups were present, these probably would have been methylated by diazomethane. However, approximately the same methoxyl content was obtained by either esterification or diazomethane methylation. Therefore, only carboxyl groups reacted with diazomethane. From this reasoning, it is probable that two aliphatic hydroxyl groups have been acetylated.

If these functional groups are subtracted from the empirical formula given above, a residue of $C_{22}H_{18}O_6$ remains. Chlorite lignin A may then be pictured as follows:

$$\left[\begin{array}{lll} CH_3O & & COOH \\ CH_3O & [C_{22}H_{18}O_6] & COOH \\ HO & & Cl \\ HO & & \end{array}\right]_x$$

To account for the large amount of oxygen present in the residue, its structure must consist at least in part of furan or pyran rings, ether linkages, and keto groups. No arrangement of three benzene rings can be made to fit the formula. It remains for future degradation studies on chlorite lignin A to determine the nature and arrangement of its "building stones."

Table IV. Elementary Analysis of Chlorite Lignin A

	Found, %	*Calculated*, %
Carbon	51.59	52.00
Hydrogen	4.64	4.67
Chlorine	5.95	5.92
Oxygen (by difference)	37.81	37.41

In an entirely analogous manner, a unit weight for chlorite lignin B may be calculated although the data in this case are not as complete. Chlorite lignin B contains 13.17% chlorine and 5.61% methoxyl, indicating that there are approximately two chlorine atoms for each methoxyl group in the smallest building unit. Again, by dividing the molecular weights of these groups by their analytical percentages, the values 540 and 553 are obtained based on chlorine and methoxyl, respectively.

When chlorite lignin B was methylated with diazomethane, the methoxyl content was approximately tripled (16.13%). This indicated that two methyl groups were introduced. If the formula given in the preceding section is used with P equal to 16.13, n equal to 1, and m equal to 2, M is 548.

Because of the similarity of this product and chlorite lignin A, and the fact that it was formed under even more drastic oxidizing conditions, it is very likely that the groups methylated by diazomethane were also carboxyls.

The acetylation data were not used because of the difficulty encountered in purifying the acetylated product.

The average of the three unit weight figures calculated above is 547.

If an attempt is made to devise an empirical formula for chlorite lignin B, the most likely is $C_{21}H_{21}O_{13}Cl_2$, which has a molecular weight of 552. Table V shows a comparison of the actual analytical values for carbon, hydrogen, and chlorine with those calculated from this formula.

Again, by subtracting the functional groups believed to be present (one methoxyl, two chlorine, and two carboxyl groups), from the empirical formula, a residue of $C_{18}H_{16}O_8$ remains. In a similar manner as before chlorite lignin B may be represented as follows:

$$\left[\begin{array}{lll} CH_3O & & COOH \\ Cl & [C_{18}H_{16}O_8] & COOH \\ Cl & & \end{array}\right]_x$$

An even higher oxygen to carbon ratio exists in this product than in chlorite lignin A. This high oxygen content cannot be accounted for except by the presence of furan or pyran rings, ether linkages, and the like.

In his work on the ultraviolet absorption spectra of lignin, Glading found that the absorption band around 280 mmu persisted in spite of such alterations in the lignin molecule as are caused by methylation, acetylation, or treatment with phenol, glycol, thioglycolic

Table V. Elementary Analysis of Chlorite Lignin B

	Found, %	*Calculated*, %
Carbon	46.04	45.75
Hydrogen	3.92	3.81
Chlorine	13.17	12.88
Oxygen (by difference)	36.87	37.70

Table VI. Material Balance for Cooks 7, 8, and 9

Cook number	*7*	*8*	*9*
Wood,[a] oven-dry, g.	1800	1800	1800
Lignin in wood, %[a]	27.65	27.65	27.65
Yield, holocellulose,[b] oven-dry, g.	1312	1316	1309
Lignin in holocellulose, %	2.16	2.44	2.57
Lignin removed, g.	470	466	464
Yield, chlorite lignin A, g.	30.3	49.5	43.8
Yield, chlorite lignin B, g.	10.5	3.1	2.0
Yield, ether-soluble A, g.	33.0	37.0	43.3
Yield, ether-soluble B, g.	141.5	132.2	178.0
Yield, ether-soluble C, g.	13.6	6.9	8.1
Total material recovered, g.[c]	228.9	228.7	275.2[d]
Recovery (based on lignin removed), %	48.8	49.2	59.3

[a] Extracted.
[b] Ash containing.
[c] Allowance must be made for increased weight due to chlorination.
[d] By letting filtrate B stand longer, a larger yield was obtained. Evidently the secondary oxidation reaction continues for some time.

acid, sodium hydroxide, or sodium hydrogen sulphide. Stamm has observed that this absorption band persists even in chlorinated lignins. Yet, chlorite lignins A and B represent modified and chlorinated lignin preparations which no longer exhibit the pronounced maximum in the absorption curve at 280 mmu. If it were only definitely known what configuration of the lignin molecule or the distribution of functional groups present is responsible for this characteristic band, this latter observation might be of value.

Sodium chlorite exercises a vigorous oxidative action on lignin. This is illustrated by the fact that the principal ether-soluble reaction product that could be identified is fumaric acid, which probably arises from the degradation of aromatic rings. Approximately one half the weight of the original lignin was unaccounted for in the total recovered material. This discrepancy was probably due in part to the evolution of gaseous reaction products such as carbon dioxide. Table VI presents a material balance for the last three cooks.

A somewhat higher percentage of recovery was obtained when an isolated lignin was used as the starting material. In the case of Indulin, about 60% was accounted for in the chlorite Indulin products and ether-soluble materials. A material balance for these experiments is given in Table VII.

From the observations made in this study, it is apparent that the lignin decomposition products are distributed in the original untreated chlorite liquor in a wide range of degree of molecularity. The highest molecular weight fraction (chlorite lignin A) is precipitable with strong acid to the extent of 9 to 11% of the original lignin. The lowest molecular weight materials are extractable with ether in an amount of 8 to 9% of the original lignin, which composes the ether-soluble A. The bulk of the material cannot be recovered by either means without further treatment. If the remaining material could be altered by further oxidation or other means to afford substances that can be recovered by precipitation or solvent extraction, the efficiency of the process would be greatly increased. One way of doing this was found in the second acidification step, in which a rather drastic oxidation reaction was initiated, and the resulting products (chlorite lignin B and ether-soluble B) reclaimed.

Table VII. Material Balance for Indulin Experiments

Experiment number,	*1*	*2*	*3*
Indulin, oven-dry	86	86	86
Yield, chlorite Indulin A	10.0	8.2	1.8
Yield, chlorite Indulin B	2.6	1.2	0.7
Yield, ether-soluble A	18.2	15.7	17.4
Yield, ether-soluble B	23.6	28.1	29.1
Yield, ether-soluble C	3.6	[a]	[a]
Total material recovered	58.0	53.2	49.0
Recovery, %	67.5	62.0	57.0

[a] Not isolated.
* All yields in grams.

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Application of Statistical Methods to Paper Testing Procedures

Part II. Reproducibility of Physical Tests

L. K. REITZ and F. J. SILLAY

A series of experiments were run to determine the reproducibility of each of six physical test methods using ten different papers. The sampling was done in such a manner that the importance of changes in physical characteristics at intervals across the machine width of the sheet and along a 50-foot section could be examined. Standard deviation for the groups of results are presented along with a comparison of inherent method variability versus variability due to position in the sheet.

THE original article (*3*) of this series on the use of statistical methods in studying paper-testing procedures was concerned with the folding endurance test. A comparison of the precision on the MIT and Schopper machines was made using several different papers. It was found that the reproducibility of the folding endurance test was poor (coefficients of variation between 20 and 30%) and it was pointed out that an observer must use discretion in interpreting test results.

A knowledge of the precision of any test procedure is necessary to distinguish between real differences among samples and those which might reasonably occur because of nothing more than inherent test method variability. Thus, the purpose of the present work was to obtain precision estimates on six more of the common physical test methods on different types of paper. The experiments were set up in such a way that, by using statistical methods, it was possible to evaluate the importance of changes in physical characteristics both along and across the machine direction of the sheet.

EXPERIMENTAL

The six physical tests which were studied are listed below: (1) penetration (Valley Size Test), (2) bursting strength (Perkins) T 403 m-41, (3) wet bursting strength (Ashcroft), (4) tear (Elmendorf) T 414 m-42, (5) stretch (Schopper) T 457 m-46, and (6) tensile (Schopper) T 404 m-47.

A 50-foot, full-width sample was obtained from a roll of each of the following papers. Each 50-foot section was then cut into 5-foot lengths, and the 5-foot sections were in turn sampled according to the plan shown in Fig. 1. This procedure resulted in tests at 10 positions across the sheet repeated at 10 positions along the sheet. Exceptions were the cross stretch and cross tensile tests where the required sample length limited the number of across positions to seven.

All tests were run at standard TAPPI atmospheric conditions of 73°F. and 50% relative humidity.

The ten papers tested represented different types of furnish and different paper machines. Thickness and basis weights were as follows:

Sample identification	*Thickness in thousandths of an inch*	*Basis weight in lb./1000 sq. ft.*
A	11.2	55
B	5.5	27
C	3.0	12
D	5.0	25
E	4.6	21
F	3.6	16
G	13.3	55
H	4.6	20
I	4.9	22
J	6.1	22

RESULTS

Because of the large volume of data (approximately 8400 results) individual test values are not tabulated. However, a variance analysis (*1, 4*) was run on each set of results and from this analysis the significance of changes in physical characteristic with position in the sheet, the components of variance, and the coefficients of variation were evaluated. These results are presented in Tables I, II, and III.

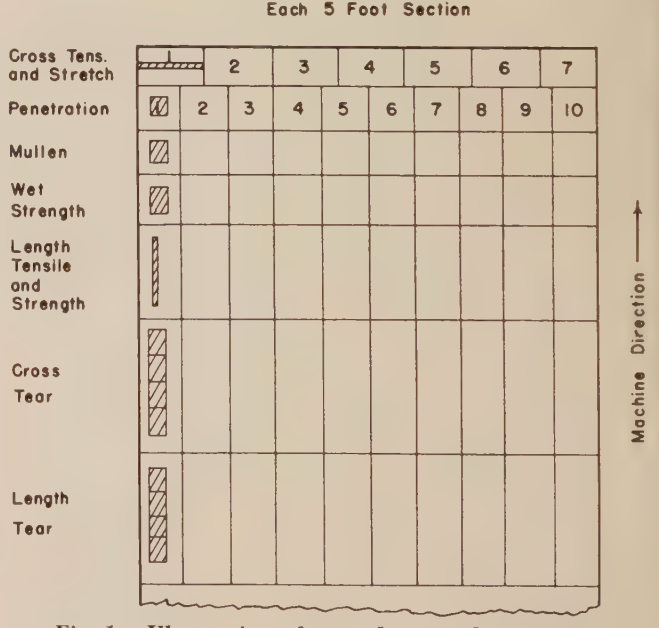

Fig. 1. Illustration of procedure used to sample

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Table I. Significance of Variability Along and Across Sheet

Paper	Penetration L	Penetration C	Bursting strength L	Bursting strength C	Wet strength L	Wet strength C	Length stretch L	Length stretch C	Cross stretch L	Cross stretch C	Length tensile L	Length tensile C	Cross tensile L	Cross tensile C	Length tear L	Length tear C	Cross tear L	Cross tear C
A	x	x	x	–	–	–	x	–	x	–	x	–	x	x	x	–	–	x
B	x	–	–	–	–	–	x	x	–	–	x	x	–	–	x	–	–	x
C	x	x	–	–	–	x	x	–	x	–	x	–	–	x	x	–	x	–
D	x	x	–	–	–	–	x	–	–	–	x	–	–	–	x	x	–	x
E	x	–	–	–	x	–	x	–	–	–	x	–	–	x	x	x	–	x
F	–	x	–	–	–	–	x	–	x	–	x	–	x	x	–	–	x	x
G	x	x	x	–	x	x	x	–	–	–	x	–	x	x	–	–	x	x
H	x	x	–	–	x	x	x	–	–	–	x	–	x	x	x	x	x	x
I	x	x	–	x	–	–	x	–	–	–	x	x	–	–	x	x	x	x
J	x	x	–	–	–	–	x	–	–	x	x	x	x	–	x	x	x	x

L = Variability along machine direction of sheet; C = Variability across machine direction of sheet; x = Variability significant; and – = Variability not significant.

Table I lists the relative significance of changes along or across the sheet. These differences in a physical characteristic are considered significant or real when there is more variability among positions than would be expected to occur because of nothing more than inherent test method error. We may say, then, that for those tests and papers marked with a "X" in Table I, differences in results were observed which must be considered to represent actual changes in the physical characteristic at various positions within the sheet.

Table II lists the relative magnitude of the portions of the total variance in results introduced by (1) random test method error, and (2) by changes in positions along and/or across the sheet. In each case the total variance is assumed to be one unit, and the portion assignable to each of the above terms is tabulated as a decimal fraction of this one unit total.

It will be noted that with one exception the random error of the test procedure is more critical than effects due to position within the sheet. This indicates that it is possible to improve materially the precision of results by merely running replicate tests within a small area of a sheet. Thus, if only a single small sample were available for test, it would be both logical and essential to run more than one test on the sample to improve the precision of the results. However, where it is practical to check more than one position from the parent lot or roll, both the variance due to position and that due to method error can be reduced by making replicate tests (*1*, *4*). For example, let us assume that the variance in a given physical test can be expressed as follows:

$$\sigma^2 \text{ total} = \sigma^2 \text{ position} + \sigma^2 \text{ method}$$

By running n replicate tests within a small area of the sheet the expression becomes

$$\sigma^2 \text{ total} = \sigma^2 \text{ position} + \frac{\sigma^2 \text{ method}}{n}$$

Table II. Portions of Total Variance Contributed by Directional Effects and Inherent Method Variability

Physical test	Inherent test variability	Along roll	Across roll	Total
Penetration	0.500	0.170	0.330	1
Bursting strength	0.730	0.226	0.044	1
Wet strength	0.746	0.142	0.112	1
Length tear	0.662	0.159	0.179	1
Cross tear	0.595	0.214	0.191	1
Length stretch	0.386	0.556	0.058	1
Cross stretch	0.562	0.168	0.270	1
Length tensile	0.585	0.398	0.017	1
Cross tensile	0.757	0.152	0.091	1

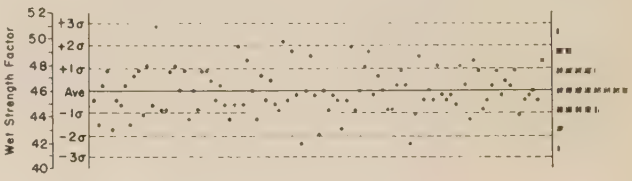
On the other hand, by selecting these n samples so that they represent different positions from the sheet or lot the expression becomes

$$\sigma^2 \text{ total} = \frac{\sigma^2 \text{ position}}{n} + \frac{\sigma^2 \text{ method}}{n}$$

Sampling by the latter method is obviously the more efficient in reducing the total variance but may also involve prohibitive testing costs. However, it becomes possible from such a study to arrive at a procedure which maintains a balance between sampling and testing costs and the desired test precision.

Table III contains the coefficients of variation found for each physical test by paper grades.

This coefficient is defined as the standard deviation expressed as a percentage of the average result (*2*). In a normal distribution it would be expected that approximately two thirds of a large number of results would fall within ±1 standard deviation from the average of all the results—about 95% should be included in the range of ±2 standard deviations from the average, and 99.7% within ±3 standard deviations. Figure 2 has been included to demonstrate this relationship.



Percentage of Total Number of Results Included in the Limits	Predicted From A Normal Distribution	Actual Results
Ave ± 1σ	68.3	70
Ave ± 2σ	95.5	94
Ave ± 3σ	99.7	100

Fig. 2. Plot of wet-strength data on paper A

The data in Fig. 2 are a plot of the wet bursting strength results on paper A. The average was found to be 46, the standard deviation 1.7, and from this the coefficient of variation given in Table III was calculated to be 3.7%. The frequency diagram was obtained by using a 1.5 unit cell size. The actual count of the percentage of occurrences which fall within ±1, 2, and 3 standard deviation limits shows good agreement with results predicted from a normal distribution.

We can say, then, that for the wet strength test on paper A approximately one out of 20 individual tests will be more than two times 3.7% (1.7 wet strength units) or 7.4% (3.4 wet strength units) above or below

Table III. Coefficients of Variation

(Standard deviation expressed as per cent of average)

Paper	Penetration	Bursting strength	Wet strength	Length stretch	Cross stretch	Length tensile	Cross tensile	Length tear	Cross tear
A	10.0	5.3	3.7	12.3	11.4	14.0	6.9	4.1	3.3
B	12.1	4.7	3.3	8.9	10.0	8.7	4.9	4.4	3.8
C	14.4	5.0	5.1	14.2	13.8	9.3	5.8	7.0	7.2
D	7.1	10.2	5.2	13.5	12.0	16.7	6.7	6.1	6.1
E	8.1	8.8	4.8	13.9	11.6	18.6	7.1	3.6	4.7
F	10.6	5.1	4.3	11.1	11.4	11.3	4.5	5.3	4.2
G	10.5	4.8	4.2	10.6	5.3	10.1	5.0	5.7	4.4
H	11.5	4.0	4.5	9.8	11.1	7.8	5.3	7.0	5.8
I	5.8	4.9	9.1	9.1	13.8	7.2	3.0	4.5	4.0
J	7.5	5.2	5.1	9.3	9.9	8.8	5.2	6.5	4.8
Av.	9.8	5.8	4.9	11.3	10.6	11.3	5.4	5.4	4.8

the average. It cannot be said, however, that even this is a fair measure of the precision of the average physical test method. From Table III it can readily be seen that this example was one of the best in the group. The coefficient of variation was low and also the variations along and across the roll were not found to be significant.

The penetration data on this same paper represent an example of one of the poorer physical tests from the group. The coefficient of variation was fairly high (10%) and it was shown by variance analysis that changes in penetration at different positions both along and across the roll were significant or real. Figure 3 is a plot of this set of results.

The letters are used in this graph to designate the positions across the roll and numbers positions along the roll. It can be seen that differences or changes across the sheet are large with results that are low at one edge of the sheet. There is also a definite tendency for results to vary with position along the machine direction of the sheet. Although the variability does not follow a fixed pattern or trend, there was a range of about 11% between the average of ten results of position 1 and position 10. (The average of the several results at each position are plotted as rectangular points.)

The coefficient of variation presented in Table III includes the variability which can be charged to the existence of these real differences among positions along and across the roll. It is realized, that because 10 is a rather small number of positions upon which to base the estimates of this variability, some of the coefficients may not be extremely accurate. However, this situation does not by any means invalidate any of the conclusions drawn from the data. Certainly the coefficients of variation are sufficiently high to warn an observer to view physical test results with a critical eye. Using the three standard deviation assurance level the precision of a single result was found to vary from about ±10% to ±60% depending upon the physical test and the paper upon which it was run.

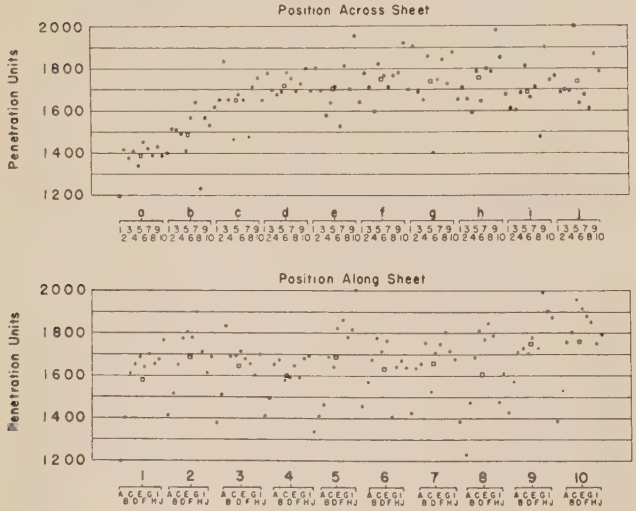


Fig. 3. Plot of penetration data on paper A

SUMMARY

The variance analyses of the data obtained on these experiments led to the following general conclusions:

1. In a large percentage of the trials there were found to be real changes in physical characteristics at different positions within the 50-foot full-width section of a roll.

The penetration test was the worst in this respect while the dry bursting strength and the wet bursting strength were best.

2. Despite the fact that these differences did exist, the largest factor contributing to variance of results was inherent test-method variability. This indicates that precision can be improved by replicates within a small sample area. The most efficient procedure, however, is one in which test samples are taken from different positions within the parent lot or roll.

3. The penetration, stretch, and tensile tests had the poorest coefficients of variation, and bursting strength, wet bursting strength, and tear tests had the best. However, the variability encountered among replicate results on any of the test methods indicates that physical test data should be examined with care. Since precision improves in proportion to the square root of the number of tests included in a reported average, an observer must consider this factor along with the inherent method variability. In any event, caution should be used to insure that undue emphasis is not placed on differences which might reasonably occur within limits of test error.

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The Chemical Analysis of Western Woods. Part III

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Four species of western hardwoods and four species of western softwoods were analyzed for ether, alcohol, and water extractives, holocellulose, lignin, methoxyl group, acetyl group, and ash content in accordance with procedures given in Part I and Part II of this series. The holocellulose and lignin procedures gave products amounting to 100% of the moisture-free weight of the extractive-free woods. Differences found in the chemical composition of hardwoods and softwoods followed the usual pattern.

THE present work is a continuation of the chemical analyses of western woods reported in two other articles of this series (*1*, *2*). In the present work, eight species of western woods were analyzed for their extractive (ether, alcohol, and hot-water), holocellulose, lignin, pentosan, methoxyl group, acetyl group, and ash content. Four hardwoods, red alder (*Alnus rubra* Bong.), tanoak (*Lithocarpus densiflorus* Rehd.), bigleaf maple (*Acer macrophyllum* Pursh.), and chinquapin [*Castanopsis chrysophylla* (Hook.) DC.], and four softwoods, lodgepole pine (*Pinus contorta* var. *latifolia* S. Wats.), Alaska yellow cedar [*Chamaecyparis nootkatensis* (Lamb.) Spach.], grand fir (*Abis grandis* Lindl.), and noble fir (*Abies procera* Rehd.), were studied.

Analytical samples for chinquapin and tanoak were taken from boards used in seasoning studies at the Oregon Forest Products Laboratory. A 1-inch long cross-sectional length was taken from each of 100 boards of each wood prior to seasoning. Most of these boards were composed of heartwood, but a few contained some sapwood. Analytical samples of the other species were cross-sectional disks that contained both heartwood and sapwood.

Table I. Extractive Content of Western Woods[a] (Quantities in Per cent)

Wood	*Ether soluble*	*Alcohol soluble*	*Hot water soluble*	*Sum of extractives*
Lodgepole pine, *Pinus contorta* var. *latifolia*	1.56	1.62	1.80	4.98
Alaska yellow cedar, *Chamaecyparis nootkatensis*	1.36	2.12	1.25	4.73
Red alder, *Alnus rubra*	0.32	2.44	1.56	4.32
Tanoak, *Lithocarpus densiflorus*	0.38	4.29	1.76	6.43
Bigleaf maple, *Acer macrophyllum*	0.30	5.12	1.62	7.04
Grand fir, *Abies grandis*	0.82	2.72	1.36	4.90
Chinquapin, *Castanopsis chrysophylla*	1.51	7.48	1.76	10.75
Noble fir, *Abies procera*	0.49	2.03	1.51	4.03

[a] Solubilities are reported on the moisture-free weight of the unextracted wood sample.

All wood samples were room dried to a moisture content of less than 10% and then reduced to particles passing a 40-mesh screen in a Wiley mill. The ground wood was stored in sealed glass jars according to TAPPI Standard T 11 m, Sampling and Preparing Wood for Analysis. Except for the holocellulose determination, which was made on the 40 to 60-mesh fraction, analyses were made on the total wood sample passing a 40-mesh screen. The samples were analyzed by the TAPPI procedures given in Parts I and II of this series. The analyses were made in duplicate by two different analysts, and the results reported in Tables I to III, inclusive, are the mean values of the four determinations.

Table II. Analysis of Western Woods[a] (Quantities in Per cent)

Wood	*Holocellose*	*Lignin*	*Pentosan*	*Methoxyl*	*Acetyl*	*Ash*
Lodgepole pine, *Pinus contorta* var. *latifolia*	72.10	28.39	15.60	4.36	1.51	0.36
Alaska yellow cedar *Chamaecyparis nootkatensis*	69.85	29.97	15.27	4.63	1.98	0.22
Red alder, *Alnus rubra*	76.36	24.14	22.81	5.51	3.15	0.23
Tanoak, *Lithocarpus densiflorus*	80.22	19.85	23.95	5.80	5.73	0.31
Bigleaf maple, *Acer macrophyllum*	76.46	23.37	17.70	4.51	4.24	0.26
Grand fir, *Abies grandis*	71.70	28.96	9.32	4.64	1.45	0.25
Chinquapin, *Castanopsis chrysophylla*	74.47	25.73	24.35	5.56	4.31	0.30
Noble fir, *Abies procera*	67.83	32.12	10.70	4.73	1.47	0.71

[a] Percentages are based on the moisture-free weight of extractive-free materials prepared according to TAPPI T 12 m-45.

It appears that the holocellulose content of each of these species has not been reported previously. In all cases, the holocellulose was white in color with the final chlorination treatment, and it darkened only slightly with the final extraction with hot alcohol-ethanolamine solution. As found previously for both hardwoods and softwoods, the sum of the lignin and the holocellulose contents is approximately 100% of the extractive-free wood. In some instances, the sum of the lignin and holocellulose contents slightly exceeds 100%. This could well be caused by the fact that the holocellulose determination was made on a screened 40 to 60-mesh

Table III. Sum of Holocellulose Plus Lignin in Extractive-Free Woods

Species	%
Lodgepole pine, *Pinus contorta* var. *latifolia*	100.49
Alaska yellow cedar, *Chamaecyparis nootkatensis*	99.82
Red alder, *Alnus rubra*	100.50
Tanoak, *Lithocarpus densiflorus*	100.07
Bigleaf maple, *Acer macrophyllum*	99.83
Grand fir, *Abies grandis*	100.66
Chinquapin, *Castanopsis chrysophylla*	100.20
Noble fir, *Abies procera*	99.95

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fraction (whereas the lignin determination was made on the whole wood fraction), by absorption of ethanolamine on the holocellulose, and by experimental errors in either determination. Residual lignin determination on the holocellulose fractions gave clear syrups with 72% sulphuric acid and virtually water-white hydrolysates, which indicated that lignin was absent.

In general, the softwoods contained more oil and resin, as represented by the ether-soluble material, than the hardwoods. Tannin, phlobaphenes, and coloring matters, as indicated by the alcohol solubles, were higher in the hardwoods. Both hardwoods and softwoods contained less than 2% water-soluble carbohydrate and miscellaneous materials.

The present analyses substantiate previously known differences in the chemical composition of hardwoods and softwoods. Lignin content is higher in softwoods than in hardwoods, and the holocellulose content is correspondingly lower in the former woods. The pentosan content and the acetic acid content of all hardwoods appears to be greater than those for the softwoods.

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Studies on Lignin and Related Products

V. Further Studies on the Oxidation of Basic Calcium Lignosulphonate with Silver Oxide and Alkali

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The effect of changing reactant ratios, reaction conditions, and type of silver oxide on the oxidation of basic calcium lignosulphonate with silver oxide and alkali to ether-soluble products was investigated. In addition to vanillic acid, vanillin, acetovanillone, and guaiacol previously reported for this reaction, a new compound, 5-carboxyvanillic acid, has been found in substantial amounts in the bisulphite-soluble and in the bicarbonate-soluble fractions.

The oxidation of basic calcium lignosulphonate (BCLS) with alkali and silver oxide employing an arbitrary ratio of 3 moles of silver oxide to one unit lignin weight of 180 was described in a recent paper (*1*). This reaction was found to yield vanillic acid as the chief oxidation product, with smaller amounts of vanillin, acetovanillone, and guaiacol as other identified products. Further exploratory experiments on this reaction indicated that the yield and nature of the products of the silver oxide oxidation of BCLS were a function of the reactant ratios and that, in all probability, the arbitrary ratios employed in the earlier work were not the best. The present paper reports a large number of experiments on the effect of varying lignin-silver oxide-sodium hydroxide ratios on the over-all yield of ether-soluble materials. These experiments comprise three series in which the amount of silver oxide was varied, maintaining the quantities of lignin and alkali constant; the alkali was varied, maintaining the lignin and silver oxide constant; and the silver oxide and alkali were varied, maintaining the lignin constant.

All experiments were performed under the same general conditions as follows:

Silver oxide was prepared by mixing aqueous solutions of silver nitrate and sodium hydroxide. The precipitated oxide was filtered, washed with water until free of nitrates, and transferred to a three-neck 1-liter flask equipped with an oil-sealed stirrer and a reflux condenser. The silver oxide was covered with 250 ml. of water and treated with stirring with the sodium hydroxide, during which the temperature of the mixture rose. If necessary, heat was applied to raise the temperature to 65°C. When the temperature reached 65°C., 25 grams of BCLS were added with continued stirring and the flask was closed with a stopper carrying a thermometer reaching below the surface of the reaction mixture. Addition of the BCLS caused a reaction and silver metal was formed. When the reaction ceased, heat was applied, and the mixture was boiled under reflux with stirring for 1 hour and allowed to cool with stirring. The cool mixture was filtered, and the silver residue was washed thoroughly with hot water. The combined filtrate and washings were acidified with dilute sulphuric acid, diluted with water to 1 liter, and extracted continuously with ether in the air-agitated extractor (*2*) for 8 hours. The ether extract was dried with sodium sulphate and distilled to dryness under reduced pressure. The residue was dried in a vacuum desiccator over calcium chloride for 24 hours and the weight of the product was recorded as ether solubles.

In the large number of experiments, every effort was made to keep conditions as identical as possible. However, variations in quantities of reactants, reaction temperatures, and other factors made this very difficult. The starting temperature of 65°C. was identical throughout, as was the length of time each mixture was refluxed and stirred. The time of extraction and the work up of the ether extracts, including the drying step, were closely controlled. Obvious factors that could not be closely controlled were: (a) losses during transfer of the washed silver oxide, (b) condition of the silver metal precipitate and resulting ease of filtration, (c) the temperature increase resulting from the initial reaction and the length of time required to reach the boiling point, and (d) the speed of ether extraction. In the last case, the speed of extraction is dependent upon the flow of air through the extractor. The flow of air, in turn, is dependent upon emulsion formation and the ease with

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which the emulsion thus formed will break after leaving the extraction chamber and entering the trap.

The results of this study are given in Table I. The first experiment was patterned after earlier work (*1*). In this experiment 25 grams of BCLS (0.076 mole of lignin) were reacted with 3 molecular equivalents of freshly prepared silver oxide and 24 molecular equivalents of sodium hydroxide. Variations from this standard experiment are reported as molecular ratios of silver oxide to lignin and sodium hydroxide to lignin, respectively. The maximum temperature column is that temperature reached by the reaction of the BCLS and alkali-silver oxide mixture before heat was applied to bring the reaction mixture to boiling. The yield column is based upon the original lignin content of the BCLS.

Many of the results are also graphed in Figs. 1 to 3. The data were plotted as results were obtained and, when minima and/or maxima were found, additional experiments were made in the range to determine more closely the exact position of the minimum or maximum. Curves 1, 2, and 3 of Fig. 1 give the variations in the over-all yield of ether-soluble products obtained by varying the ratio of silver oxide while maintaining the sodium hydroxide : lignin ratio constant at 18.0, 19.2, and 21.0, respectively. Curves 4, 5, and 6 of Fig. 2 give the same data for sodium hydroxide : lignin ratios of 22.4, 24.0, and 48.0, respectively. Fig. 3 gives the variation in the yield of product obtained by varying the sodium hydroxide : lignin ratio while maintaining the silver oxide : lignin ratio constant at 3.0.

It should be noted that these experiments were made under arbitrary conditions in order to obtain a large amount of information in a short time. An 8-hour extraction was employed, although it is known that longer extraction times are necessary for complete removal of ether solubles. In addition, total ether solubles are reported. No attempt was made to fractionate the extracts and only the weights of total extracts are compared. It is entirely possible that the amount of vanillic acid might be much greater in one of the smaller ether extracts than in one of the larger fractions that is essentially a dark semisolid in appearance.

Several facts became evident from this series of experiments.

Under all conditions of oxidation, the yield of product appeared to pass through a maximum and a minimum with increasing silver oxide before the highest yields were obtained. This is easily explained because the characteristics of the products obtained at the two maxima are entirely different and it is evident that the product first formed is ultimately converted to the product obtained with the larger amount of silver oxide.

The data of this series also indicate that the hitherto employed 3 moles of silver oxide per lignin unit weight of 180 was somewhat lower than that required for maximum conversion of the lignin to ether-soluble products. Under all conditions of alkali concentration, the highest yields of ether solubles were obtained at much higher silver oxide:lignin ratios.

The highest yield of ether solubles was obtained with a silver oxide : lignin ratio of 3.75 and a sodium hydroxide:lignin ratio of 21.

The amount of alkali does not appear to be an important factor as far as total yield is concerned (see Fig. 3). However, the amount of alkali does have an important bearing on the reaction. The condition of the silver residue is influenced by the alkali concentration. The less alkali used, the more finely divided and difficult to filter becomes the silver residue. In experiment 20, in which only 25 grams of sodium hydroxide were employed, the silver residue remained suspended in the alkaline solution as a colloid and passed through the fine sintered glass funnel used for removing the silver in all experiments. The boiling point of the reaction mixture was also dependent upon the alkali concentration.

All ether-soluble products obtained from reaction mixtures employing like quantities of silver oxide ap-

Table I. Oxidations of Basic Calcium Lignosulphonate[a] with Silver Oxide and Alkali

Experiment	*Molar ratio silver oxide: lignin*	*Molar ratio sodium hydroxide: lignin*	*Maximum temperature, °C.*	*Boiling temperature, °C.*	*Yield, %*
1	3.0	24.0	88	106	19.2
2	1.5	24.0	73	106	19.2[b]
3	6.0	24.0	76	106	15.8
4	0	24.0	58	106	10.6[c]
5	2.25	24.0	76	106	17.6
6	4.5	24.0	71	106	24.1
7	3.75	24.0	74	106	24.0
8	4.0	24.0	95	106	23.2
9	2.0	24.0	72	108	16.0
10	5.0	24.0	74	106	24.7[d]
11	2.6	24.0	77	107	16.5
12	5.25	24.0	81	105	23.2
13	5.5	24.0	75	105	22.1
14	3.0	12.0	81	103	19.0
15	3.0	36.0	72	110	18.1
16	3.0	30.0	82	108	18.4
17	3.0	32.0	77	108	17.6
18	3.0	42.0	75	112	19.6
19	3.0	16.0	84	102	17.9
20	3.0	8.0	75	101	15.8[e]
21	3.0	18.0	89	105	19.7
22	3.75	18.0	86	104	16.1
23	4.0	18.0	92	104	20.3
24	4.5	18.0	92	104	15.2
25	5.0	18.0	89	104	24.3
26	3.5	18.0	92	104	17.8
27	2.25	18.0	83	105	9.0
28	1.5	18.0	78	105	11.6
29	3.0	48.0	70	114	22.2
30	3.75	48.0	71	115	22.2[f]
31	5.0	48.0	76	114	25.2
32	4.5	48.0	83	114	22.1
33	3.0	21.0	83	105	20.7
34	3.75	21.0	83	105	28.2[g]
35	5.0	21.0	83	105	19.6
36	4.5	21.0	91	105	27.5
37	4.0	21.0	94	105	24.3
38	3.5	21.0	89	105	21.5
39	3.75	21.0	84	105	26.7
40	2.0	21.0	89	105	12.9
41	1.5	21.0	75	105	15.0
42	2.25	21.0	82	105	14.4
43	1.0	21.0	72	104	9.8
44	3.75	19.2	76	105	26.0
45	3.5	19.2	77	105	23.6
46	4.0	19.2	76	105	17.6
47	5.0	19.2	79	105	21.7
48	2.25	19.2	83	105	14.9
49	1.5	19.2	76	105	17.3
50	3.75	22.4	75	105	20.0
51	3.5	22.4	76	105	21.9
52	4.0	22.4	78	105	21.7
53	5.0	22.4	78	105	18.3
54	2.25	22.4	82	105	12.5

[a] The BCLS was of spruce origin and was kindly furnished by the Marathon Corp. of Rothschild, Wis. The product had the following percentage analysis: moisture, 11.6; CH_3O on solids, 9.0. Assuming lignin to contain 14.5% methoxyl, the lignin content of the "as-received" BCLS becomes 54.7%.

[b] This product was a viscous liquid. It contained 6.5% vanillin.

[c] This product was a liquid.

[d] This product contained 0.8% vanillin.

[e] The silver residue remained suspended and a large percentage passed through the sintered glass funnel during the filtration step.

[f] This product contained 0.7% vanillin.

[g] This product contained 0.8% vanillin.

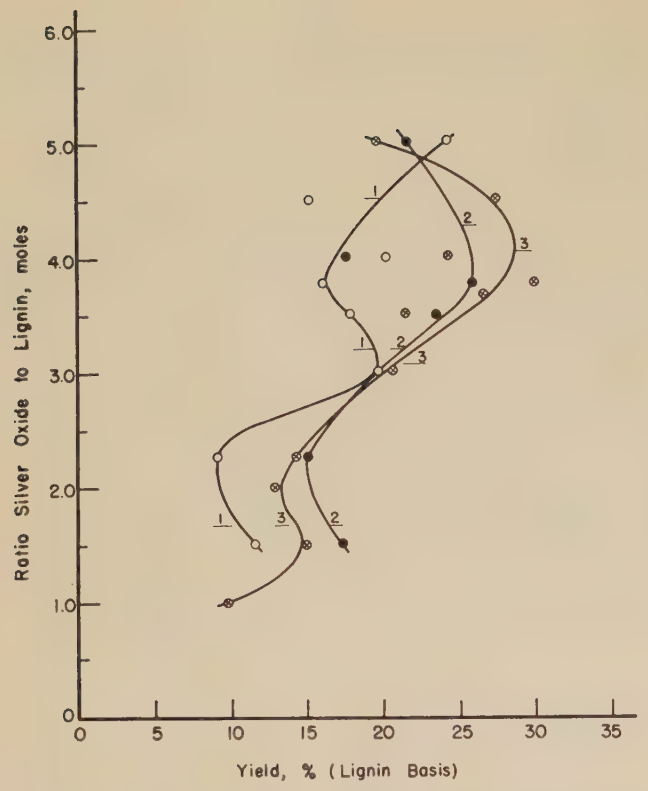


Fig. 1. Yields with various silver oxide : lignin ratios
Curve 1. Sodium hydroxide : lignin ratio constant at 18.0; Curve 2. sodium hydroxide : lignin ratio constant at 19.2; and Curve 3. sodium hydroxide : lignin ratio constant at 21.0.

peared much the same, regardless of the amount of alkali used. The products obtained from reactions employing silver oxide: lignin ratios of 3 or more appeared as light tan solids presumably containing large percentages of vanillic acid, whereas the products from reactions employing silver oxide: lignin ratios of less than 3 were much darker in color and were often oily or semi-solid. These products usually possessed phenolic odors.

Vanillin analyses were made on four representative products (experiments 2, 10, 30, and 34). These data, expressed as percentages of the ether-soluble products, are given as footnotes in Table I. It is evident that, under conditions of low silver oxide ratios, the vanillin yield is much higher than under other conditions, but under no circumstances are vanillin yields very high in the presence of silver oxide or silver. This is to be expected in view of the known instability of vanillin in the presence of an alkaline solution and silver metal or silver oxide (*3–5*). Furthermore, it was found impossible to recover or even analyze for a large proportion of vanillin added to synthetic or natural BCLS-silver oxide reaction mixtures.

LARGE-SCALE EXPERIMENTS

Having determined conditions for maximum yields of ether solubles, large-scale experiments were made employing conditions for obtaining maximum yields. Experiment 60 was modeled after experiment 34 in Table I, which gave the highest yield of ether-soluble product. Silver oxide was freshly prepared from 678 grams of silver nitrate, filtered, washed free of nitrates, and transferred to a 5-liter three-neck flask. The silver oxide was covered with 1500 ml. of water and treated with 390 grams of solid sodium hydroxide with stirring. When the temperature reached 65°C., 150 grams of

Table II. Fractions Obtained in a Large-Scale Oxidation Simulating Experiment 34

(Experiment 60)

Fraction	*Yield, % (lignin basis)*
Bisulphite solubles	13.9
Bicarbonate solubles	14.1
Alkali solubles	1.5
Neutrals	1.9
Total	31.4

BCLS were added. Reaction took place immediately and the temperature rose to 78°C. The mixture was heated to boiling under a reflux with stirring for 5 hours and allowed to cool. The mixture was worked up and fractionated by the one-step sulphuric acid procedure outlined in the earlier paper (*1*). The results are given in Table II.

The bisulphite-soluble fraction in experiment 60 was almost as large as the bicarbonate-soluble fraction. In this experiment, however, both fractions were light colored solids. The large bisulphite fraction is explained by data from several experiments on the isolation of vanillin and vanillic acid from mixtures of the two. These data showed that, when a mixture of vanillin and an excess of vanillic acid was extracted with 21% sodium bisulphite solution, the bisulphite extract yielded an impure vanillic acid instead of vanillin. Therefore, the actual vanillic acid yield of experiment 60 is higher than that indicated by the size of the acid fraction.

The conditions of experiment 60 were repeated in experiment 61 but, in this experiment, the reaction mixture was worked up by the two-step sulphur dioxide-sulphuric acid method detailed in the earlier paper (*1*). The data for experiment 61 are given in Table III.

The over-all yield by the two-stage method was considerably greater than that by the one-stage method. The yield of bicarbonate solubles from the sulphur dioxide solution was especially high, again indicating that lack of prior sodium bisulphite extraction results in higher bicarbonate solubles. The bicarbonate-soluble fraction in this experiment was a very light powdery solid containing over 85% vanillic acid.

EFFECT OF REACTION CONDITIONS

Dilution

Having determined that the reactant ratios of experiment 34 gave the highest yields of ether solubles by the procedure employed in all these studies, several experiments were made to determine whether changes in reaction procedure would affect the yields. In all experiments, the reactant ratios and method of work up employed in experiment 61 were used.

Experiment 62 employed a reaction mixture with twice as much water as the standard. The increased

Table III. Fractions Obtained in a Large-Scale Oxidation Simulating Experiment 34

(Two-Step Work up—Experiment 61)

Fraction	*SO_2 Acidified, %*	*H_2SO_4 Acidified, %*	*Total, %*
Bisulphite solubles		6.9	6.9
Bicarbonate solubles	23.6	0.7	24.3
Alkali solubles	1.8	0.4	2.2
Neutrals	2.0	1.1	3.1
Total	27.4	9.1	36.5

dilution caused a lower boiling point of the reaction mixture and resulted in lower yields.

In experiment 63 only 1000 ml. of water were employed instead of 1500 ml. as in the standard. The temperature of boiling of this reaction mixture was 107°C., and the yields were essentially those of experiment 61. Apparently, the concentration of the reaction mixture does not greatly affect the results obtainable by the oxidation of BCLS under identical silver oxide-alkali-lignin ratios, provided the temperature of boiling remains above 105°C.

Type of Silver Oxide

A few experiments were made on the generation of silver oxide *in situ*. The reactant ratios of experiment 61 were employed. In experiment 64 a mixture of 150 grams of BCLS, 549 grams of sodium hydroxide, and 700 ml. of water was stirred and, at 65°C., a solution of 678 grams of silver nitrate in 800 ml. of water was added over a period of approximately 5 minutes with vigorous stirring. The temperature began to rise almost immediately, and soon reached the boiling point (108°C.). The reaction mixture was boiled under a reflux for 5 hours, allowed to cool with stirring, and worked up by the two-step procedure. The yields in this experiment were approximately 20% less than the standard.

In order to obviate side reactions which might be caused by the excess of silver oxide present at the beginning of the reaction, experiment 65 was performed in the same manner except that the silver nitrate solution was added over a period of 45 minutes. Under these conditions the yields approximated those of the standard experiment.

It should be noted that, in the last two experiments, it was necessary to concentrate the extract from the sulphuric acid-acidified solution under an atmosphere of sulphur dioxide to minimize the nitration reaction taking place [see (*1*)]. Benzene may be employed instead of ether in order to obviate the nitration reaction with almost the same result, although longer extraction times are required. The use of benzene instead of ether gives good yields of bisulphite, bicarbonate, and alkali-soluble materials, but not of the neutral fraction.

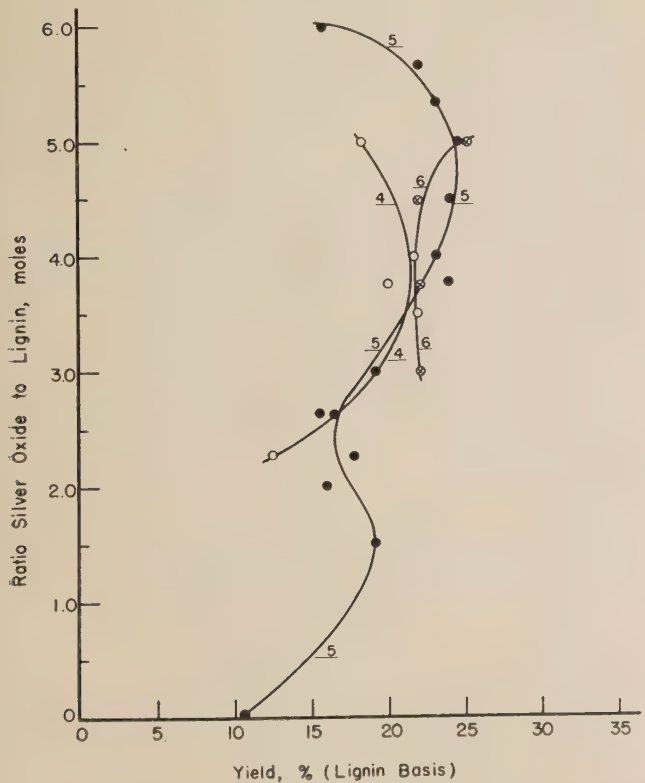


Fig. 2. Yields with various silver oxide : lignin ratios
Curve 4. Sodium hydroxide : lignin ratio constant at 22.4; Curve 5. sodium hydroxide : lignin ratio constant at 24.0; and Curve 6. sodium hydroxide : lignin ratio constant 48.0.

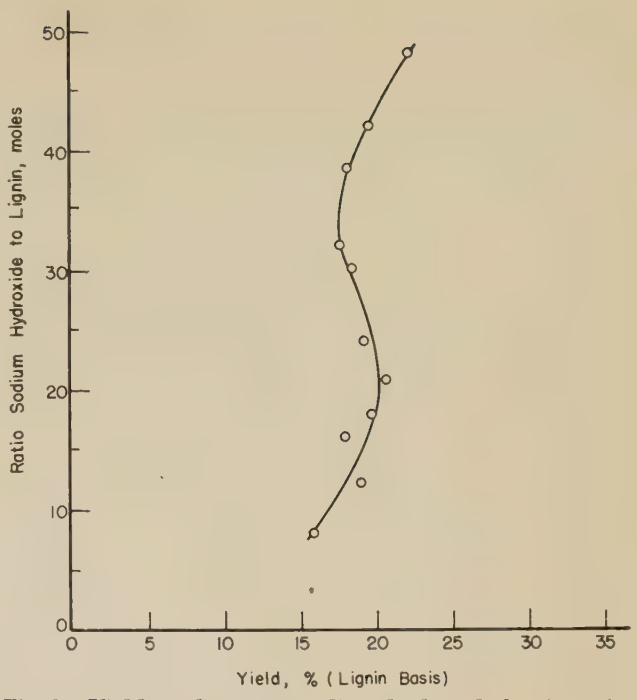


Fig. 3. Yields with various sodium hydroxide:lignin ratios
Silver oxide : lignin ratio constant at 3.0.

Experiment 66 was made in a manner identical with that of experiment 61 except that dry commercial silver oxide was used in place of freshly prepared and moist silver oxide. The products of reaction were obtained in substantially the same amounts, thus indicating that the freshness of the silver oxide is not a determining factor in the oxidation reaction.

In earlier work on the oxidation of vanillin with silver oxide and alkali (*3*), it was demonstrated that the silver metal resulting from the reduction of the oxide could be reoxidized with potassium permanganate to give a silver oxide admirably suited for reuse to give an essentially quantitative transformation of vanillin to vanillic acid. This silver oxide recovered by means of permanganate was very black and finely divided, and did not resemble silver oxide prepared by mixing aqueous solutions of silver nitrate and sodium hydroxide. Other evidence led us to believe that this material was not ordinary silver oxide, but rather a higher oxide or a mixture of higher oxides of silver. Experiment 67 was performed to see whether the recovered black oxide of silver behaved as ordinary silver oxide in the oxidation of BCLS. After the reaction was complete, the silver oxide still appeared to be unreduced. Work up by the two-step procedure gave only about 30% of the fractions obtained in the analogous standard experiment employing regular silver oxide. In addition, the nature of the fractions was different. The data of experiment 67 is more evidence that the recovered oxide of silver is not ordinary silver oxide.

It is obvious that the total yields of the experiments reported in this paper are not as high as those of the

silver oxide oxidation of BCLS reported earlier (*1*). The reason for this lies in the nature of the BCLS. In a comprehensive study on the effect of the original pulping conditions upon the oxidizability of lignosulphonate materials, we have found that the amount of, and distribution of the fractions of the silver oxide oxidation of a lignosulphonate material are functions of the original cooking conditions and the after treatment given the liquor. The BCLS employed in this series of experiments was prepared about 6 years after the BCLS used earlier, and apparently its pretreatment was somewhat different. The details of the comprehensive study on the effect of the original pulping conditions on the oxidation products will be published in the future.

ISOLATION OF REACTION PRODUCTS

The isolation of vanillic acid, vanillin, acetovanillone, guaiacol, eicosanol, and 4-nitroguaiacol from the silver oxide-BCLS reaction mixture was reported in the earlier paper (*1*). In addition to these compounds, a new compound, 5-carboxyvanillic acid, has been found in the bisulphite-soluble and in the bicarbonate-soluble fractions.

5-Carboxyvanillic Acid from the Bisulphite-Soluble Fraction

The light brown crystalline bisulphite-soluble fraction from a one-step work up experiment (experiment 60) (5.0 grams) was covered with 100 ml. of dry ether, heated to boiling under a reflux, and filtered. The clear solution was treated with 50 ml. of dioxane. The still clear solution was distilled very slowly from a warm water bath to remove the ether. Upon cooling crystals separated from the dioxane solution. On standing, more crystals separated. These were filtered and washed with a little cold dioxane. The yield of almost colorless crystals melting at 276–277°C. was 2.0 grams. The combined filtrate and washings were concentrated to yield 0.2 gram of similar material. Repeated recrystallization from water raised the melting point to 281°C. This product was identical with the 5-carboxyvanillic acid isolated from a similar fraction obtained on oxidation of *Torulopsis utilis*-fermented sulphite spent liquor with silver oxide (*6*). The total yield of 2.2 grams corresponded to 39% of the original bisulphite-soluble fraction and, in this case, represented 5% of the orginal lignin.

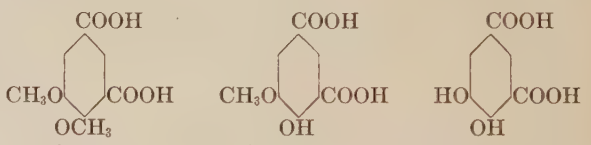
The presence of 5-carboxyvanillic acid in the bisulphite-soluble fraction is easily explained. 5-Carboxyvanillic acid, being a dibasic acid, is quite soluble in water. Because 21% sodium bisulphite solution is the first aqueous solution employed for the fractionation of the ether-soluble material, it naturally extracted the water-soluble dibasic acid. Under the conditions of isolation of 5-carboxyvanillic acid, the other bisulphite-soluble materials are not removed. The other materials, such as vanillin and acetovanillone, are very soluble in dioxane and, therefore, do not crystallize from solution when the ether is removed from the dioxane-ether solution.

5-Carboxyvanillic Acid from Bicarbonate-Soluble Fraction

The bicarbonate-soluble fraction from the extract of the sulphur dioxide-acidified reaction mixture of experiment 61 was subjected to the lead acetate procedure for the isolation of vanillic acid as described earlier (*1*). The precipitated lead salts were washed with cold water, treated with an excess of dilute sulphuric acid, and extracted with ether. The ether was dried with sodium sulphate and distilled to yield 2.8 grams of acidic material. This residue was covered with cold anhydrous dioxane and filtered. The residual crystalline material was recrystallized from water to yield 1.8 grams of 5-carboxyvanillic acid as colorless needles melting at 280–281°C. The yield of 5-carboxyvanillic acid in this fraction amounted to 2.1% (on the original lignin basis).

Although 5-carboxyvanillic acid has not been isolated as such from lignin oxidation mixtures, there is evidence in the literature that a structure which would yield 5-carboxyvanillic acid is present in lignin. Freudenberg, Janson, Knopf, and Haag (*7*) treated lignin with caustic soda at 210°C. and methylated the total product directly with dimethyl sulphate. From this mixture they isolated 2 to 4% of isohemipinic acid, indicating that either 5-carboxyvanillic acid or 5-carboxyprotocatechuic acid was present in the original reaction mixture.

Richtzenhain (*8*) treated hydrochloric acid lignin with 20% hydrogen peroxide in the presence of barium carbonate, isolated the acids giving insoluble barium salts, methylated with diazomethane, fractionated the methyl esters, and obtained 0.1% isohemipinic acid and 0.04% 5-carboxyvanillic acid as their methyl esters. Hydrolysis with alkali yielded the pure acids. Again, this experiment indicates the presence in a lignin oxidation mixture of either 5-carboxyvanillic acid or 5-carboxyprotocatechuic acid, but no evidence to indicate either specifically.



Isohemipinic acid — 5-Carboxyvanillic acid — 5-Carboxyprotocatechuic acid

The finding of 5-carboxyvanillic acid is helpful in explaining the fact that a silver oxide oxidation of a given lignin material will yield more vanillic acid than a nitrobenzene oxidation will yield vanillin. 5-Carboxyvanillic acid is probably formed as an intermediate in the transformation of at least a part of the lignin into vanillic acid. This intermediate then loses carbon dioxide to form vanillic acid.

Studies on the synthesis of 5-carboxyvanillic acid and its degradation to vanillic acid will be reported at a later date.

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The Operation and Application of a Rotary Gravity Filter

J. K. PERKINS

The Waco filter was invented in Sweden in 1939 and tested in this country in 1948 on a number of different types of paper mill waters. It may be used as a catchall on industrial effluents where large volumes are discharged, thereby reducing stream pollution from pulp and paper mills, textile mills, chemical industries, etc. In many cases the filter can deliver effluents to the sewer with the efficiency of a sedimentation system using chemical flocculents. In pulp, paper, and wallboard mills the filter is used within the forming machine system to recover fibers in the white water and it renders filtrates normally suitable for showers and overflow to the sewer. As a water filter suspended solids are removed with the efficiency of a straight sand filter. The Waco filter is a rotary gravity filter covered with a traveling wire cloth similar to that on a paper machine. The filtration is not done by the wire alone, but by a thin filter medium formed in advance on the wire before it enters the liquid containing suspended solids to be removed. The preformed coat of filter medium on the wire does the filtration and no influent passes through the bare wire. In most cases the filter medium is a layer of wood pulp.

Among the more important elements in the manufacture of pulp and paper is the treatment of water required in the process. Treatment is normally required at three points in the system: first, raw water for mill use; white water for reuse; and, now becoming more important, the treatment of mill sewer effluents for recovery of valuable material and for stream improvement.

This paper will show how a new type filter will fit each of the requirements mentioned, its operation, and the performance which can be expected from it.

During the last two years in the United States and the last ten years in Sweden, this new filter has been under trial and development. The principle used is the known efficiency of a mat of cellulose fiber when used as a filter medium. Such an arrangement was usable only batchwise until the Waco filter, Fig. 1, was designed in Sweden in 1936. This new filter handles 400 to 1500 g.p.m. when operated as a paper machine saveall (depending upon the amount and character of the suspended solids in the white water handled). It will handle 1000 to 5000 g.p.m. when operating on mill sewers as a catchall and will handle 5000 g.p.m. or 7,200,000 gallons per day as a raw water filter. The filter has proved itself to be a very flexible and versatile machine in the removal of suspended solids from liquids.

PRINCIPLE OF OPERATION

This filter operates on the principle used in the laboratory for the separation of suspended solids from a liquid. This type of filtration incorporates the use of a fibrous mat (normally a cellulose filter paper or an asbestos mat in a Gooch crucible), through which the liquid containing the suspended solids is drained or pulled by vacuum. The suspended solids are initially retained on or in the mat of cellulose because their size is larger than the capillaries or voids in the layer of fibrous material.

The Waco filter accomplishes this same function continuously by precoating its main filtering drum with a layer of wood pulp before the wire comes in contact with the liquid to be filtered. This layer of fiber is continuously formed and then removed automatically after use. The freeness and amount of suspended solids in the feed determine the ultimate capacity. The capacity is also a function of the speed of the wire; therefore, the wire speed is regulated to the characteristics of the material in the influent to obtain a desired volume of flow.

The filter incorporates established mechanical pulp handling methods in a new arrangement, Fig. 2. It has an open cylinder filtering drum, a sheet forming cylinder, and a traveling wire. A standard wire guide actuates one wire supporting roll; the other supporting roll functions as a couch. Thus the machine is simple and is easily operated and maintained by men familiar with standard pulp and paper equipment.

The main filtering drum does not have a fixed cover but is intended only to support the traveling wire face and has solid rings at each end to form a seal with the wire. The cylinder has a double deckle at the discharge end, one deckle strap over the other on separate supports. Clean water is kept at a slightly higher head

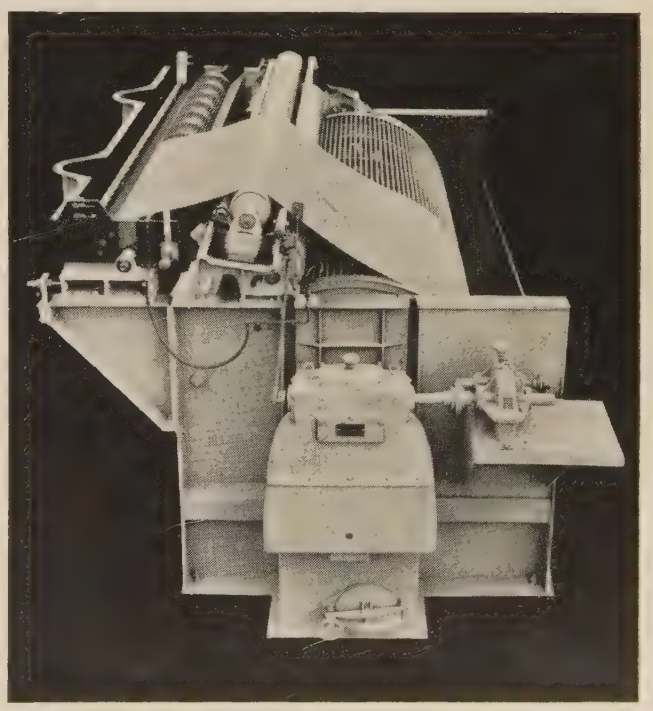


Fig. 1

J. K. Perkins, Sales Engineer, Improved Paper Machinery Corp., Nashua, N. H.

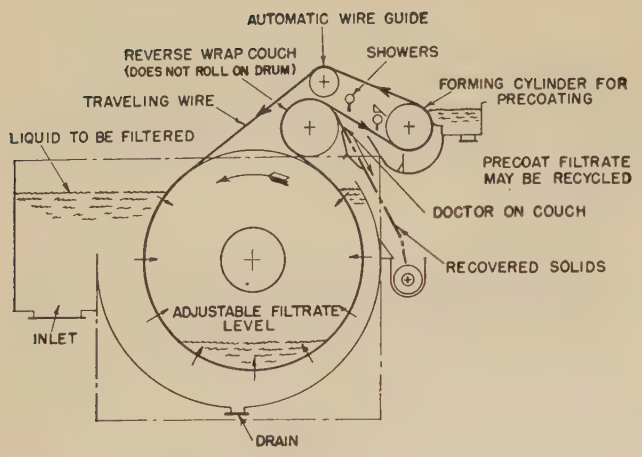


Fig. 2. Diagram of filter

between them to prevent any leakage from the vat into the filtrate.

Only the main cylinder is driven and it is geared to a variable speed drive to give the required speed range.

The main vat is of conventional design. When used as a water filter a slightly larger headbox with baffles to cut down turbulance at the inlet lip is required.

The precoat system provides the means by which slush pulp is formed into a continuous filter sheet. It is made up of a small, open cylinder with a top feed headbox. The lower part of the vat is used to collect the filtrate from the prefilter cylinder; from here it returns to the fan pump. Pulp or machine furnish from a stuff box is added to the suction of this fan pump which delivers it back to the headbox. Excess white water from this system may be piped to the main vat for refiltering.

Acting only as a vehicle to carry the filter sheet is an "endless" traveling wire. It laps around the precoat cylinder at the point where the sheet is formed and laps around the main cylinder covering the area submerged in the filter vat. Between the precoat cylinder and the main cylinder the wire is supported by the guide roll; on its return to the precoat cylinder it laps around a couch roll.

When operating on white water, the filtrate discharge of the machine is regulated by a butterfly valve which is controlled by the level in the vat. If for any reason the level in the vat falls, the discharge valve begins to close. This has the effect of raising the level inside the cylinder and decreases the flow through the sheet. If the vat level rises, the valve opens allowing the level to fall inside the cylinder increasing the filtering gradient. This feature establishes uniform flow from the filter.

On raw water the control is usually on the inlet. The possible and practical means of connecting the filter to automatically adjust itself to the situation are too numerous to mention here, as each would be engineered to the particular situation in the mill.

OPERATION

As the wire passes the lip in the precoat headbox, the filter sheet begins to form. Almost immediately after the wire with its coat of stock emerges from the surface, it leaves the forming cylinder and passes over the guide roll and begins its lap on the main cylinder slightly above the maximum level in the vat. As the pulp mat enters the liquid, the filtration velocity is very slow and accelerates as the gradient increases. The precoat mat and the deposited material are held in place on the wire, while in the vat, by the pressure differential across the filter face.

As the filter mat completes its travel around the main filtering drum, it passes between the wire and the couch roll, where excess water is pressed out by the tension on the wire. This produces discharge consistencies of between 8 and 14% air dry. As the traveling wire leaves the couch roll, the pulp adheres to the couch, from which it is subsequently removed by a doctor. The wire then travels on under a wire cleaning shower and is again ready for a new layer of precoat material The discharge pulp, along with its recovered suspended solids, may be returned to the process, as in the case of a paper machine saveall, or discarded or downgraded in the case of a water filter. If the amount of pulp needed for water filtration is high, a means has been devised for washing this pulp and returning it for reuse. Normally, the pulp consumption for water filtration is less than 100 pounds per 24 hours and does not warrant recovery.

Data have been collected from the units now operating as savealls and water filters, and from it certain trends have been established.

So far, it has been impossible to establish any definite correlation between the dependent and the independent variables on the basis of individual runs. The characteristics of the influent, such as freeness and particle size, distribution, etc., (which cannot be controlled or evaluated), are of considerable importance, and these characteristics vary between different pulp and paper stocks.

Wire Speed and Capacity

The easiest trend established was the corresponding change in capacity obtained with a change in the wire speed. This requires little explanation; it is simply the fact that the flow through the sheet decreases gradually as the suspended matter collects on and in the mat. The retarded rate of flow is usually the same for any

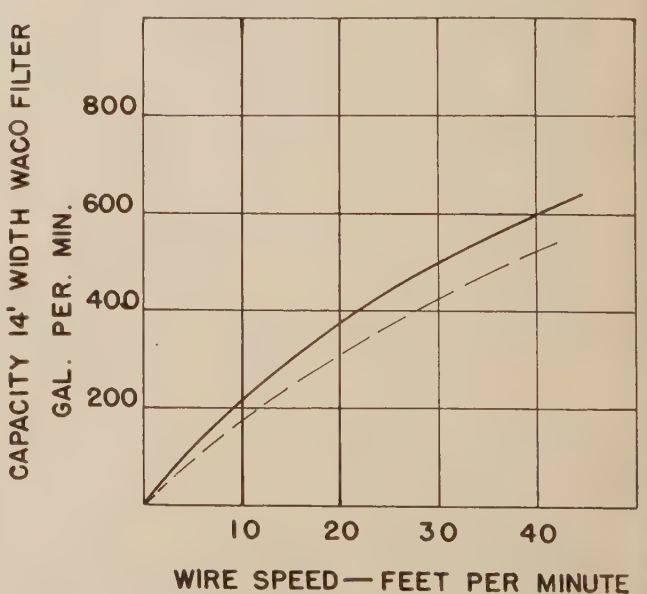


Fig. 3. Speed of filter vs. capacity on tissue paper machine white water

particular material. Consequently, if more filter area is exposed per unit of time, the capacity is greater. The ratio between a change in wire speed and the resulting change in capacity is not directly proportional. This can be seen in Fig. 3. This curve is an average of 33 test runs made while filtering paper machine white water in a tissue mill. The results obtained during these tests averaged:

Influent to filter to 9.3 pounds per 1000 gallons.
Effluent from filter, 0.177 pounds per 1000 gallons.
Average mat weight, 0.0103 pounds per square foot.
Recovery efficiency, 98.1%.
Type of precoat: blended groundwood and chemical fiber for machine furnish, unjordaned.
Source of white water: combined excess from several machines.
Nature of white water: short groundwood and chemical fiber, with some flour, and no clay or chemicals.

The dotted curve in Fig. 3 represents the prediction made by special leaf tests on a similar white water which

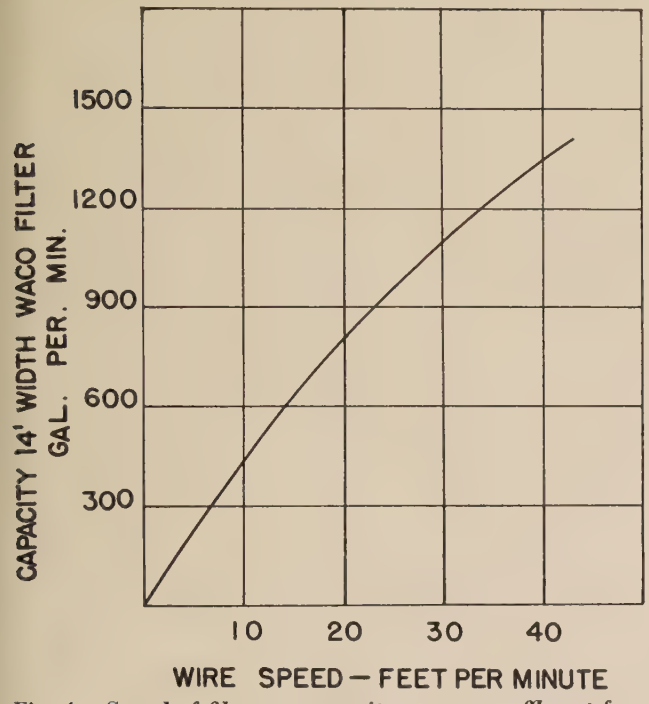


Fig. 4. Speed of filter vs. capacity on sewer effluent from tissue mill

led to the installation of the large machine in the tissue mill.

The effect of wire speed on capacity can also be seen in Fig. 4 when filtering sewer effluent from the same tissue mill. The capacity is much greater for a given wire speed as the amount of material to be removed is so much less. This curve is a plot of values from test runs when the suspended solids in the feed to the filter varied between 1.40 and 1.65 pounds per 1000 gallons. The other data collected for the curve averaged as follows:

Influent to filter, 1.55 pounds per 1000 gallons.
Effluent from filter, 0.07 pounds per 1000 gallons.
Mat weight, 0.013 pounds per square foot.
Recovery efficiency, 95.4%.
Recovery settleable solids, 100.0%.
Type of precoat: groundwood and chemical fiber for machine furnish, unjordaned.
Source of feed water: effluent from vacuum savealls with some beater water added.

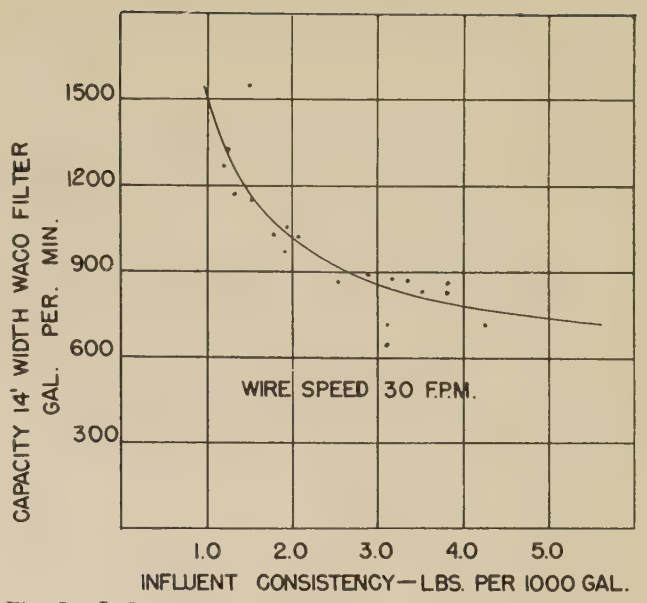


Fig. 5. Influent consistency vs. capacity on tissue white water

Nature of feed water: very short fibers, mostly groundwood flour, no clay or chemicals.

Couch Breaks

During the tests on machine white water, and while operating at the maximum speed of 40 f.p.m., an increase in capacity occurred at a time when the consistency of the influent was 19.6 pounds per 1000 gallons (due to a wet end break). There was no change in the filtrate consistency, while the recovery efficiency rose to 99.1%. This removed any doubt about the ability of the machine to handle couch breaks. The increased proportion of long fibers more than made up in added freeness for the increase in feed consistency.

Influent Consistency and Capacity

After a considerable amount of data had been collected, it was found that a definite trend was established between the influent consistency and the capacity. Figure 5 shows the points are scattered due to other

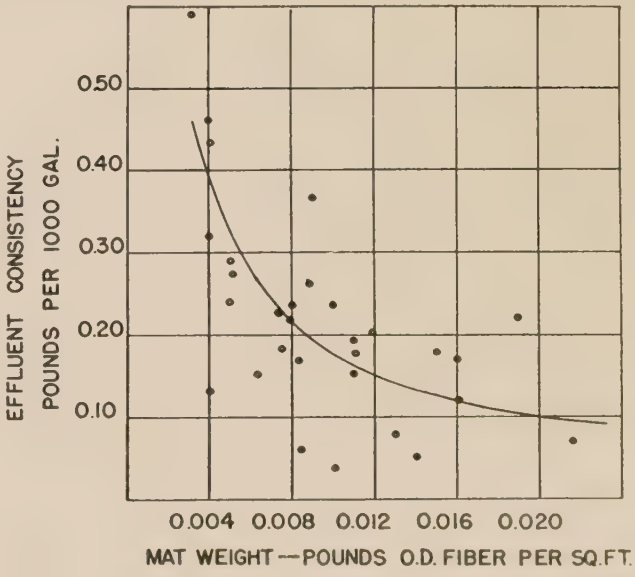
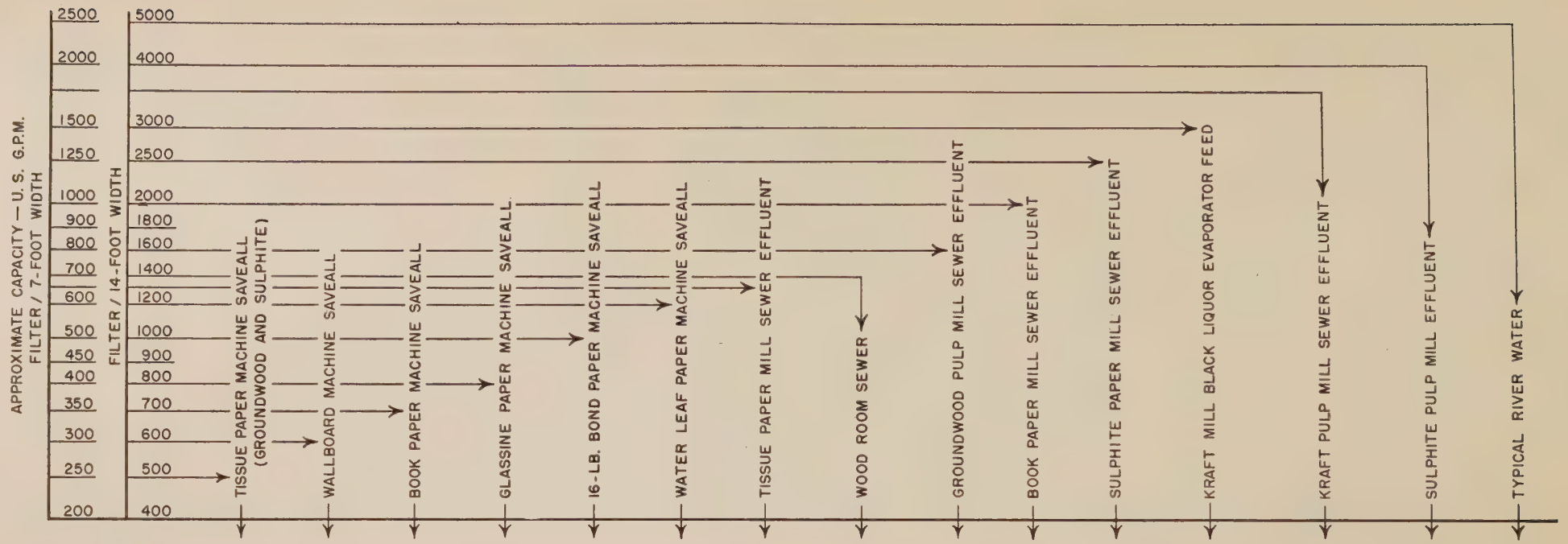


Fig. 6. Mat weight vs. filtrate clarity on tissue white water



	Tissue paper machine saveall (groundwood and sulphite)	Wallboard machine saveall	Book paper machine saveall	Glassine paper machine saveall	16-lb. bond paper machine saveall	Water leaf paper machine saveall	Tissue paper mill sewer effluent	Wood room sewer	Groundwood pulp mill sewer effluent	Book paper mill sewer effluent	Sulphite paper mill sewer effluent	Kraft mill black liquor evaporator feed	Kraft pulp mill sewer effluent	Sulphite pulp mill effluent	Typical river water
Suspended solids in liquid before filter, lb./1000 gal.	9.8	12.5	10.4	4.06	19.7	6.11	2.83	5.2	1.53	2.13	3.27	50.5 p.p.m.	1.7	1.9	29.13 p.p.m.
Nature of solids	Groundwood flour	Fibrous	72% Clay	Fibrous	8% Clay	Fibrous	Groundwood flour	Bark flour	Fibrous	Clay and fiber	70% Clay	Refiner fines	Considerable short fibers and fines	Mostly long fibers and screenings	Fine dirt
Suspended solids in liquid after filter, lb./1000 gal.	0.21	0.30	0.74	0.05	0.12	0.05	0.04	0.41	0.01	0.14	0.96	3.5 p.p.m.	0.05	0.02	2.0 p.p.m.
Wire speed using a 14 ft. width filter, f.p.m.	35	25	15	30	32	30	36	25	30	25	10	4.7	4.5	2.0	0.5
Type precoat mat	⅕ Groundwood pulp	Wallboard stock	Bleached sulphite	Machine furnish	Machine furnish	Machine furnish	½ Groundwood pulp	Sulphite screenings	Groundwood pulp	Deink pulp	Sulphite screenings	Kraft	Recovered fiber	Recovered fiber	Sulphite
Precoat pulp required, tons/24 hr.	4	9	3.8	3.5	2.9	4.0	4.0	1.5	2	1.5	1.0	0.7	0.8	0.4	0.05
Temperature, °F.	96	111	90	90	90	90	85	45	80	85	80	Up to 190	75	75	45
pH	5.0	5.4	4.5	5.0	4.5	5.0	6.0	6.8	5.7	4.7	4.52	,,	9.0	7.6/no cooking acid	...

This table is used to indicate the probable size filter required and wire speed for typical services, based on checks of actual installations and tests of numerous materials. It may be necessary or desirable to run check tests to establish the exact size and evaluate the materials of construction which should be used for mechanical parts

variables, but a trend can be seen. Values from tests at 30 f.p.m. are shown. Note that the trend becomes more pronounced at the lower limits of the consistency.

Wire Speed and Clarity

When water which contains a considerable amount of suspended solids is filtered, the mat fills up early in the cycle and the deposited material quickly becomes the filter medium. Conversely, if the water or liquid contains only a limited amount of foreign substance, such as raw water, the mat fills slowly, and the clarity of the filtrate depends more on the mat itself. This explains why the speed of the wire, within limits, does not influence the clarity of the filtrate on white water and why raw water may show a better effluent from dirty feed water than from comparatively clean water at a particular wire speed. Results of tests made while filtering raw river water, demonstrates this last statement:

Wire speed, f.p.m.	*Influent, p.p.m.*	*Effluent, p.p.m.*	*Discharge flow,*[a] *g.p.m.*
5.75	2.64	1.05	4500
5.75	1.29	0.26	4500
5.75	1.71	0.40	4500
5.75	163.80 (fine dirt added)	trace	4500
5.50	1.58	0.13	4500
7.20	29.13 (fine dirt added)	trace	4500

[a] Maximum supply pump capacity.

Notice that when the fine dirt was added to the inlet, the filtrate showed only a trace of suspended matter. Apparently the dirt reduced the size of the capillaries or voids in the sheet, producing a cleaner effluent.

Mat Weight and Clarity

Tests made on a filter installed as a machine saveall in a wallboard mill, demonstrated that the weight of the mat and its density directly affect the clarity of the filtrate.

When operating with a normal weight mat of machine furnish, the filter delivered a filtrate consistency of around 2 pounds per 1000 gallons. The wallboard furnish was very coarse for this service so the mat weight was tripled, and the filtrate consistency dropped to 0.5 pounds per 1000 gallons. In order to better this figure, a suction box was placed under the wire following the guide roll. A denser sheet resulted with some loss in flow, but the filtrate averaged less than 0.3 pounds per 1000 gallons which was acceptable for discharge to a local stream.

As this wallboard saveall was in production, no variations in data could be taken to establish trends; but when the mat weights from the data taken on the tissue machine were correlated, it was found that a definite trend was established between the mat weight and the clarity of the filtrate. This may be seen in Fig. 6.

No correlation of the data to show that the thickness of the mat influenced the capacity, except at the extreme limits, was possible.

Special Leaf Tester

Under certain circumstances it is desirable to make a leaf test on a water or suspension before making recommendations for a particular service. A specially designed apparatus in which the action of the filter is simulated is used. This tester allows a very close determination to be made of the capacity and clarity obtainable under the same conditions that exist at the mill. The accompanying diagram, Fig. 7, shows the results obtained from a few of these leaf tests and from installations now in operation. In running these tests all over the country, it was found that in many cases the white water from a particular weight and grade of paper varied in characteristics between different mills.

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Products Obtained by Destructive Distillation of Douglas-Fir Bark

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Several runs on the destructive distillation of Douglas-fir bark were made at temperatures varying from 745 to 910°F. and at distillation times of 2 to 10 hours. The yields of charcoal, insoluble tar, pyroligneous acid, and noncondensable gases were determined in each instance. The pyroligneous acid was analyzed for acetic acid, acetone, methanol, and dissolved tar. Volatile and nonvolatile portions of insoluble tar were determined by steam distillation, and these two were analyzed for acids, phenols, and neutrals. A proximate analysis of the charcoal was made. The yield of charcoal is much higher than that reported on the previous work done on Douglas-fir waste wood and sawdust and other woods, while the yields of byproducts compared favorably with that reported on Douglas-fir wood.

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Douglas-fir (*Pseudotsuga taxifolia* (Poir.) Brit.) bark is one of the major by-products of the lumber industry of the Pacific Northwest. It is now largely used as a fuel or is disposed of in most instances in refuse burners. Calculated on the basis that 20.8 cubic feet of bark having a specific gravity of 0.40 are available from each thousand board feed of logs cut in Oregon and Washington sawmills, the potential annual supply of such bark is about 1,500,000 tons. Better utilization of Douglas-fir bark would be of considerable importance to this region.

EXPERIMENTAL PROCEDURE

The apparatus used for the destructive distillation of Douglas-fir bark is shown in Fig. 1. In each run, about $2^1/_2$ to 3 pounds of bark cubes (1 by 1 by 1 inches) were carbonized in an iron retort heated by a muffle furnace

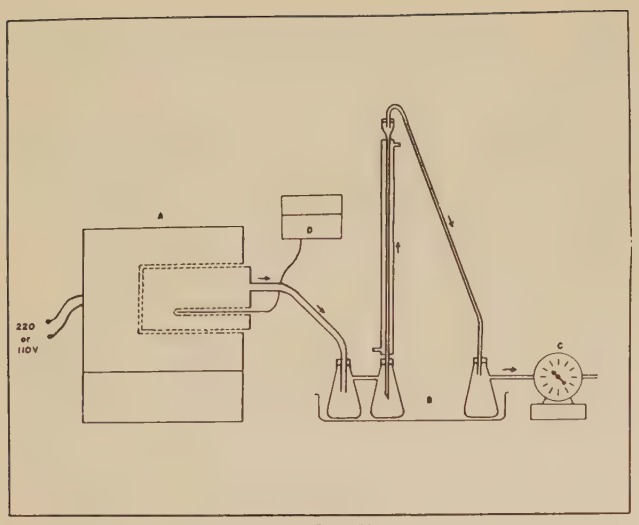

Fig. 1. Destructive distillation apparatus

(A) that could be operated at temperatures up to 1850 °F. Volatile products were passed through a condensing train (B), and noncondensable gases were measured with a wet gas meter (C). Temperature was measured with an iron-constantan thermocouple connected to an automatic potentiometric recorder (D). Following each run, the retort was cooled, and the condensate and charcoal were studied. The condensate contained two distinct parts: insoluble tar and pyroligneous acid, the former floating on the latter.

Pyroligneous acid

Acetic acid was determined by titration against standard sodium hydroxide, using a Beckmann pH meter for determination of the end point. A distilled sample of pyroligneous acid was used for the determination (*8*). The tar left in the distillation flask was assumed to be dissolved tar. Acetone was determined by Messinger's method (*6*). Methanol was determined by the estimation of methoxyl groups, using the modified Zeisel method (*13*). The test sample used for the determination of acetone and methanol was prepared by neutralizing the distilled pyroligneous acid with sodium hydroxide and redistilling it.

Insoluble Tar

Following removal of charcoal particles by filtration, the insoluble tar was subjected to the scheme of analysis shown in Fig. 2.

Charcoal

Moisture, volatile matter, ash, fixed carbon, apparent specific gravity, and true specific gravity were determined by A.S.T.M. methods (*1*).

PRESENTATION OF DATA

The product recoveries in the 14 runs made during the investigation are shown in Table I.

Table I. Product Recoveries from the Destructive Distillation of Douglas-Fir Bark, Based on the Oven-dry Bark Weight

Run number	*Maximum temperature, °F.*	*Length of run, hr.*	*Charcoal, %*	*Condensate, %*	*Gas, %*
1	745	6.00	54.3	...	...
2	815	6.50	49.1	...	...
3	815	7.50	56.0	...	...
4	795	8.84	46.6	33.95	19.50
5	780	7.00	49.7	29.50	20.80
6	770	7.75	45.5	35.78	18.72
7	770	7.50	49.8	35.98	14.22
8	745	6.50	47.4	37.28	14.75
9	800	8.75	49.7	30.70	19.60
10	870	10.17	46.1	36.98	16.92
11	870	2.00	46.3	36.48	17.32
12	885	2.25	49.7	36.58	13.72
13	870	2.33	43.3	35.60	21.10
14	910	2.50	42.0	36.60	21.40

The experiments can be divided conveniently into two groups: Runs 1 through 10 were subjected to lower average temperatures and longer distillation periods than runs 11 and through 14. The average product re-

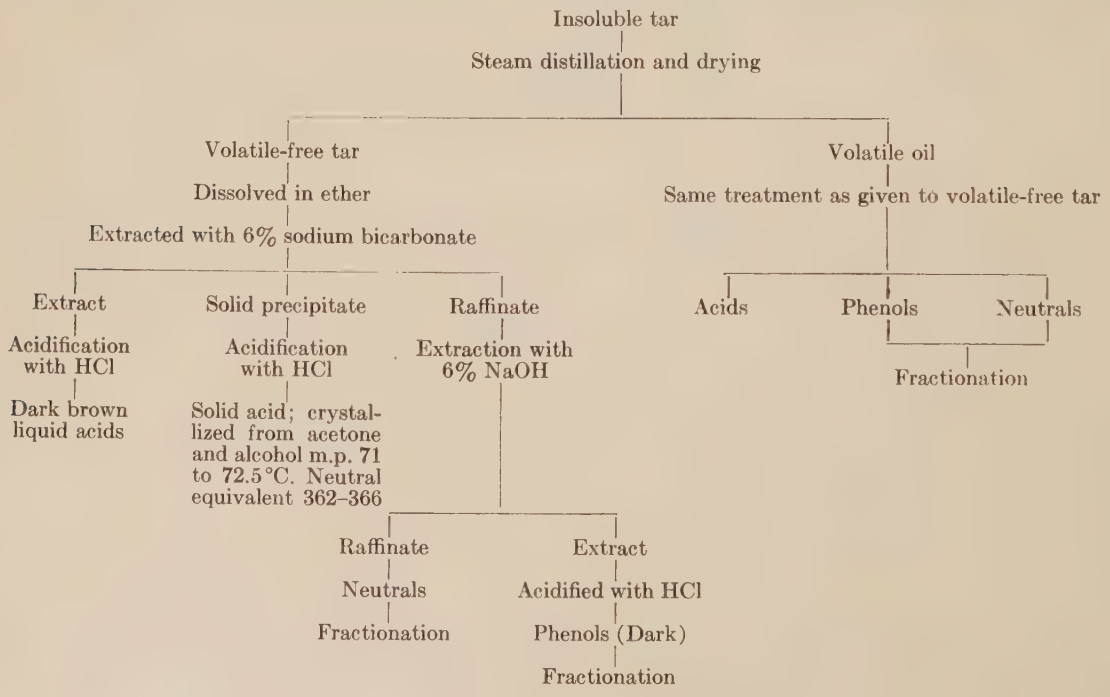

Fig. 2. Scheme for the analysis of insoluble tar

Table II. Average Product Recoveries from Two Groups of Destructive Distillation Runs on Douglas-Fir Bark, Based on the Oven-Dry Bark Weight

Run numbers	*Charcoal, %*	*Condensate, %*	*Gas, %*
4 through 10	47.8	34.4	17.8
11 through 14	45.3	36.3	18.4

coveries from these two groups of runs are presented in Table II.

Table III gives the composition of pyroligneous acid obtained by the destructive distillation of Douglas-fir bark.

Table III. Percentage Composition of Pyroligneous Acid from Douglas-Fir Bark, Based on the Oven-Dry Bark Weight

Composite samples from run numbers	*Acetic acid, %*	*Methanol, %*	*Acetone, %*	*Dissolved tar, %*
4, 5, and 6	0.781	...	0.570	3.28
11 and 12	0.648	0.348	0.357	3.06
13 and 14	0.886	0.412	0.364	1.82

Steam distillation of the insoluble tar gave the results shown in Table IV.

Table IV. Composition of Insoluble Tar from the Destructive Distillation of Douglas-Fir Bark

Composite sample from run numbers	*Nonvolatile tar*		*Volatile oil*	
	Based on oven-dry bark weight, %	*Based on insoluble tar weight, %*	*Based on oven-dry bark weight, %*	*Based on insoluble tar weight, %*
11 and 12	11.0	78.8	2.8	20.2
13 and 14	9.9	76.8	3.0	23.2

The nonvolatile and volatile portions of the insoluble tar were analyzed in accordance with the scheme outlined in Fig. 2, and a complete analysis of the products obtained in runs 13 and 14 is given in Table V.

Analyses of Douglas-fir bark charcoal gave the results shown in Table VI.

During the analysis of the insoluble tar, a solid precipitate that was soluble in alcohol, acetone, and ligroin was obtained. The material was crystallized from both 95% alcohol and acetone. Its melting point ranged from 71 to 72.5°C., and its neutral equivalent varied from 362 to 366. This nearly corresponds to a C_{24} acid. Lignoceric acid ($C_{23}H_{47}COOH$) has a melting point of 81°C. and a neutral equivalent of 368.5.

The fractionation of the various acids, phenols, and neutrals did not yield pure compounds; no derivatives could be prepared for identification.

Table V. Percentages of Products Obtained in Runs 13 and 14, Based on Oven-Dry Douglas-Fir Bark Weight

Product	*Percentage*
Charcoal	42.90
Noncondensable gas	21.18
Acetic acid	0.89
Acetone	0.36
Methanol	0.41
Nonvolatile tar	
Acids	1.31
Phenols	5.42
Neutrals	3.15
Volatile tar	
Acids	0.35
Phenols	1.18
Neutrals	1.45
Dissolved tar	1.82
Water and losses	19.58
Total	100.00

Table VI. Analyses of Douglas-Fir Bark Charcoal, Moisture-Free Charcoal Basis

Component	*Composite sample from runs 1 through 10*	*Composite sample from runs 11 through 14*
Ash, %	1.39	1.21
Fixed carbon, %	81.49	81.26
Volatile combustibles, %	17.10	17.51
Moisture content, %	1.68	2.40
Apparent specific gravity	0.15	0.13
True specific gravity	1.09	1.32

DISCUSSION

Comparison of the results obtained in the present work with that done on the carbonization of Douglas-fir waste wood (*2*, *3*), Douglas-fir sawdust (*11*, *12*), and the destructive distillation of other woods indicates the following:

1. The yield of charcoal in the destructive distillation of Douglas-fir bark is higher than that reported for such distillation of Douglas-fir waste wood (*3*), Douglas-fir sawdust (*12*), and all other woods (*5*, *9*, *10*).
2. The charcoal obtained from bark has volatile matter and fixed carbon comparable to that obtained from wood, though it has more volatile matter than the char obtained from Douglas-fir sawdust that was carbonized at a much higher temperature (1250°F.). Higher temperatures during carbonization give charcoal with lower volatile matter (*7*).
3. The yields of acetic acid, methanol, and acetone are slightly higher than those reported for Douglas-fir wood but much lower than those reported for hardwoods.
4. The yield of tar is greater for Douglas-fir bark than that reported for Douglas-fir wood, and it is of the same order as that obtained from hardwoods.
5. The yield of noncondensable gases is of the same order as that obtained from wood.

The results obtained in the present investigation very nearly bear out the conclusion reached by Bergstroem (*4*) that bark gives a higher yield of charcoal and tar and a lower yield of acetic acid and methanol.

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The Chemistry of the Sulphite Pulping Process and the Evaporation of Its Waste Liquor

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The reactions between lignin and acid in the sulphite pulping process are reviewed. Also mentioned are the effect of the acid on the sugars that are dissolved from the wood by the cooking liquor and the formation of other aldehydic substances. The chemistry of the process is of great importance in the utilization of the waste liquors and also in the problem of evaporation and subsequent burning. Because of the economic conditions peculiar to Sweden, the development of a satisfactory method of evaporation of sulphite waste liquors became necessary during the war. The causes for the formation of calcium sulphate, which causes scale on the surfaces of the evaporator, were investigated, and two systems, currently in use in Sweden, the Rosenblads and the Ramens systems, are outlined.

As all know, it was Benjamin C. Tilghman, who, in 1866, found that a white fibrous pulp is obtained when wood is heated with a water solution of sulphurous acid or, still better, a solution of calcium bisulphite. Only a few years later two other pioneers, namely the Swedish engineer, C. D. Ekman, and the German, A. Mitscherlich, succeeded, independently of each other and possibly without knowledge of Tilghman's work, in developing a technical process for the manufacture of sulphite pulp.

CHEMISTRY OF THE SULPHITE PULPING PROCESS

During the infancy of the sulphite industry, there was little interest shown in a desire to study the reactions responsible for the dissolution of the "incrusting materials" in the wood, and it took nearly two decades before actual research on this interesting problem began. In 1890 Pedersen announced that the waste liquor, obtained from the delignification of wood with calcium bisulphite, contained calcium salts of organic sulphonic acids. The well-known sugar chemist, Tollens, together with his associates Lindsay and Streeb, came to the conclusion, in 1892, that this amorphous substance which could be precipitated from sulphite waste liquor by alcohol, hydrochloric acid, or lead acetate, contained methoxyl groups and must be considered as a sulphonic acid derivative of lignin.

It took three more decades before the reaction between the lignin in the wood and the sulphite cooking acid was further investigated. These studies, that began in the author's institute in Abo in the middle of the nineteen twenties made it possible to obtain a definite picture of the over-all process and to explain many of the phenomena accompanying sulphite cooking. Even today, research in this field is devoted to completing this picture with details especially concerning the mechanism of the chemical reactions. Developments in this research are naturally intimately connected with a better understanding of the general chemistry of lignin.

The most essential point in the theory of the sulphite cooking process, as far as could be ascertained from our original research, is that the release of lignin takes place in two steps. The first step is the sulphonation of lignin in the solid state, that is, the formation of a solid lignin sulphonic acid in the wood. The second step is the dissolution of this solid lignin sulphonic acid.

The first step, the formation of the primary solid lignin sulphonic acid, can be easily demonstrated if the cooking with bisulphite solution is allowed to take place at a relatively high pH, for example, with a mixture of sodium bisulphite and sodium sulphite at a pH of 6. Under these conditions, even at normal cooking temperature (for example, 130°C.), only a very limited amount of lignin is dissolved. The largest amount of the lignin remains in the wood and is sulphonated there very rapidly until 1 sulphur atom per 3 to 4 methoxyl groups has been introduced into the lignin; after this point the sulphonation proceeds very slowly. With sodium bisulphite cooking at a pH of about 4.5, the sulphonated wood reaches a limiting value of 1 atom of sulphur per 2 methoxyl groups. However, even at this pH, the dissolution is very slow.

In order to proceed to the second step, the actual dissolution, one might assume that the solubility of lignin is an exclusive function of the degree of sulphonation and that a certain concentration of sulphonic acid groups in the lignin molecule would be necessary to make it water soluble. However, this is not the case. It can be shown that the dissolution of the solid lignin sulphonic acid is controlled by the hydrogen ion concentration. Lignin sulphonic acid dissolves from sulphonated wood, for example, by warming with an acid buffer solution. The speed of dissolution increases with decreasing pH. The lignin sulphonic acid exists in the sulphite pulp as the calcium salt. It exchanges the calcium for hydrogen ions by washing with acid. From such hydrogen ion saturated pulp Kullgren was able to dissolve the lignin sulphonic acid by merely warming with water. This observation fitted, as we could prove, excellently, with our understanding of the dissolution process. We found that the solid lignosulphonic acid surprisingly enough is to a very high degree dissociated and that the solid lignin sulphonic acid functions itself, during cooking, as a supplier of hydrogen ions. The total hydrogen ion concentration around the solid acid is dependent upon the ratio between the metal and hydrogen ions in the surrounding cooking acid.

It appeared possible to classify this hydrogen ion

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catalysis of the dissolution of lignin sulphonic acid as a hydrolytic process. We could, for example, assume that the lignin, by means of a keto group, was joined by an acetallike bond to some carbohydrate component and that a hydrolytic splitting of this acetal bond was a prerequisite for the dissolution of the lignin sulphonic acid. Another alternative is as follows. Lignin exists in wood in a relatively high molecular state where the large molecules are built up of smaller components linked together by acetal bonds. A hydrolytic splitting of such bonds, catalyzed by hydrogen ions, should lead to a breaking down of the large molecule into smaller components which would be soluble.

The existence of carbonyl groups in lignin, masked by their acetal character, has also been assumed by B. Holmberg. He explained the dissolution of lignin in alcoholic mineral acids as a conversion of the original lignin carbohydrate acetal into the lignin acetal of the added alcohol. Furthermore, Kullgren had been able to show that solid lignin sulphonic acid can be dissolved from sulphonated wood with alcohol instead of water after the metal cations have been exchanged for hydrogen ions, and that the alcohol group is taken up by the lignosulphonic acid.

These concepts concerning the sulphonation and dissolution of lignin, that have been discussed briefly, lead also to an explanation of the undesired condensation of lignin sulphonic acid in pulp to a dark insoluble product known as "burnt cook." The danger of this condensation becomes acute when a high local acidity appears in the fibers. This condition can be brought about by several different causes.

It is known, for example, that sulphurous acid diffuses considerably more rapidly in wood than calcium ions. A too rapid increase in the reaction temperature during the beginning of the cooking process can therefore lead to a significant formation of free solid lignin sulphonic acid in which the strongly acid sulphonic groups function as condensation agents for lignin molecules that lie within its sphere of influence. In order to avoid such condensation effects, it is of great importance to impregnate the wood with cooking acid before increasing the temperature to its maximum value.

During the formation and dissolution of lignin sulphonic acid there occurs, in all probability, a competition between the hydrolytic and the condensation reactions. Under proper conditions, not too high acidity or temperature, the condensation reactions can be retarded. It has been shown that it is possible to delignify wood with only sulphurous acid without the addition of any base if the temperature is kept low enough. This, however, naturally necessitates a much longer reaction time. Lignin sulphonic acid becomes less susceptible to condensation factors, such as high temperature and low pH, with increasing degree of sulphonation. This circumstance can be very easily explained by the fact that the groups which can be condensed are identical to the groups which can be sulphonated. The more these groups are replaced by sulphonic acid groups, the more resistant the molecule becomes to condensation.

One can, consequently, speak of the competition between the sulphonation and condensation processes as well as the competition between hydrolytic dissolution and condensation.

These relationships reflect themselves, naturally, in the properties of lignin sulphonic acids formed under different cooking conditions. If one sulphonates wood, for example, at a relatively high pH, for example with sodium sulphite at a pH of 5 to 6, there is only a relatively small amount of lignin dissolved. At least 60% of the total lignin content remains in the wood as solid sodium lignin sulphonate. The degree of sulphonation here is low. The product contains only 1 sulphur group per 3 to 4 methoxyl groups. This acid is, therefore, very susceptible to condensation, and its hydrolytic dissolution, which can be accomplished with water after the sodium ions have been exchanged for hydrogen ions, must be done at a relatively low temperature, preferably not over 80 to 90°C. At higher temperatures it tends to condense to insoluble products. On the other hand, if we examine some dissolved lignin sulphonic acid prepared under normal technical conditions, we find that it contains 1 sulphur group per 2 to 2.5 methoxyl groups. Such a relatively highly sulphonated lignin sulphonic acid is considerably more stable. It does not, for example, condense during the cooking process, although it is there exposed to high temperatures and, in the case of cooking acids with low base content such as are used in the preparation of rayon pulps, also high acidity, often a pH of around 1. The tendency to condense is, nevertheless, even noticeable in such highly sulphonated products. It manifests itself, for example, in the fact that rayon pulp waste liquors contain a larger amount of high molecular, or so-called α-sulphonic acids, than liquors from strong pulp cooks. It is these α-lignin sulphonic acids, possibly together with their condensation products, that cause the dark coloration of the liquor at the end of the cooking. The so-called β-lignin sulphonic acids, which are distinguished by their lower molecular weight, are, in contrast in the α-acids, dialyzable through cellophane-membranes and not precipitatable by organic nitrogen bases. These β-lignin sulphonic acids are especially rich in sulphur and surprisingly stable toward acids.

The author has, so far, discussed the sulphonation of lignin, to a large extent, without taking into consideration the specific chemical mechanisms. It is, however, obvious that we must attempt to describe the sulphite process in terms of its chemical constitution and to express it in the form of a chemical formula. It is also obvious that the development of this attempt is intimately connected with the development of our knowledge regarding lignin's chemical structure in general. The author is convinced that further study of the course of sulphonation will definitely contribute to the solution of the problem of lignin's constitution.

It is to be expected that the reaction between lignin and bisulphite is of a specific nature and that a closer study of this reaction can lead to conclusions regarding the chemical structure of the reacting groups. Furthermore, it has been shown that the conversion of lignin to lignin sulphonic acid constitutes a relatively minor change in the lignin molecule. This assumption can be illustrated by some results obtained in the author's laboratory with the help of optical methods.

A few years ago P. Lange took up the study of ultraviolet absorption in very thin cross sections of spruce and obtained the following results: the fiber walls

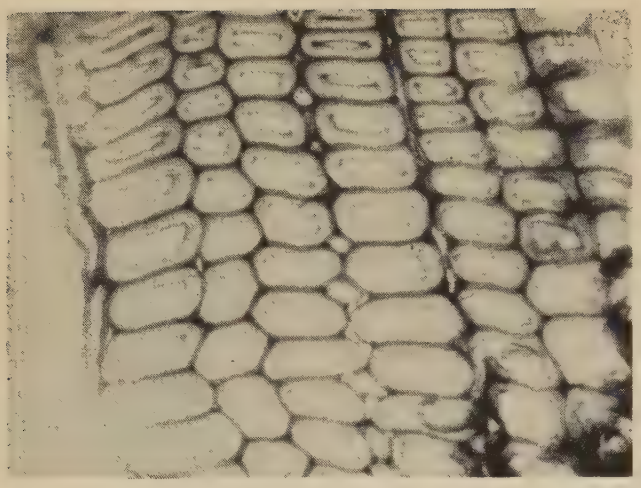


Fig. 1. Ultraviolet microphotograph of a spruce cross section in its original state

contain an ultraviolet absorbing substance whose absorption spectrum shows a decided maximum at 2800 A. This substance therefore is of an aromatic nature. By quantitative comparison of the ultraviolet absorption of wood with that of certain model substances, Lange has been able to estimate the lignin content in spruce at 27 to 29% which corresponds very well to the value obtained by chemical means. This serves also as proof of the fact that the total lignin exists in the wood as an aromatic compound. From the distribution of ultraviolet absorption across the fiber walls, it has been estimated that about 70% of the total amount of lignin is concentrated in the outer layer of the cell, the so-called "primary wall," while the remaining is distributed in the "secondary wall."

Lange has also studied the changes in ultraviolet absorption occurring at different phases of a sulphite cook and has thereby been able to follow the dissolution of lignin.

Figure 1 shows an ultraviolet microphotograph of a spruce cross section in its original state. The strongly absorbing part, which is rich in lignin, lies between the cell walls. The weaker absorbing cell walls and the nonabsorbing lumina can also be seen. After preheating with sulphite cooking acid for $5^1/_2$ hours, during which time the temperature was gradually increased to 120°C., the middle lamella can be seen to have been strongly attacked, and rows of cells have been separated from each other under the influence of the microtome (Fig. 2). After eight hours, when the temperature has reached its maximum value of 135°C., the fibers are relatively easy to separate (Fig. 3). After nine hours, the absorption is very weak and the fibers are held together only in spots (Fig. 4). After eleven hours of cooking, delignification and reduction to fibers is practically complete (Fig. 5).

These figures can also indicate something about the route by which the cooking acid reaches and attacks the lignin. Even early in the cook the middle lamella seems to be strongly attacked by the cooking acid. Special determinations of the changes occurring in lignin distribution during the cook have confirmed this view. Thus it appears that the cooking acid reaches the lignin easier by penetrating along the middle lamella than by diffusing from the lumen through the cell walls.

By photographing the cross sections at different wavelengths Lange was able to obtain the absorption curves for the lignin remaining in the cells at different times during the sulphite cook. The most important result of these measurements is that the general character of absorption of the original lignin is not changed during sulphonation. This may be interpreted that no deep-seated change in the light-absorbing system of the molecule occurs on sulphonation.

A similar result was obtained in the author's laboratory by the study of ultraviolet absorption of isolated lignin sulphonic acids in water solution.

Gunhild Aulin-Erdtman has also measured the ultraviolet absorption of lignins (*Tappi* 32, no. 4; 160–166 (April, 1949). The ultraviolet absorption for a lignin sulphonic acid containing only 1 sulphur atom per 3.2 methoxyl groups was determined. This acid was then subjected to a renewed sulphite cooking whereby two new preparations were obtained, one with (1) S per 2.1 OCH_3 and one extremely high sulphonated product with 1 S per 1.2 OCH_3. From the resulting curves it can be seen that the introduction of additional sulphonic acid groups in a low sulphonated product does not cause any important changes in the chromophoric system.

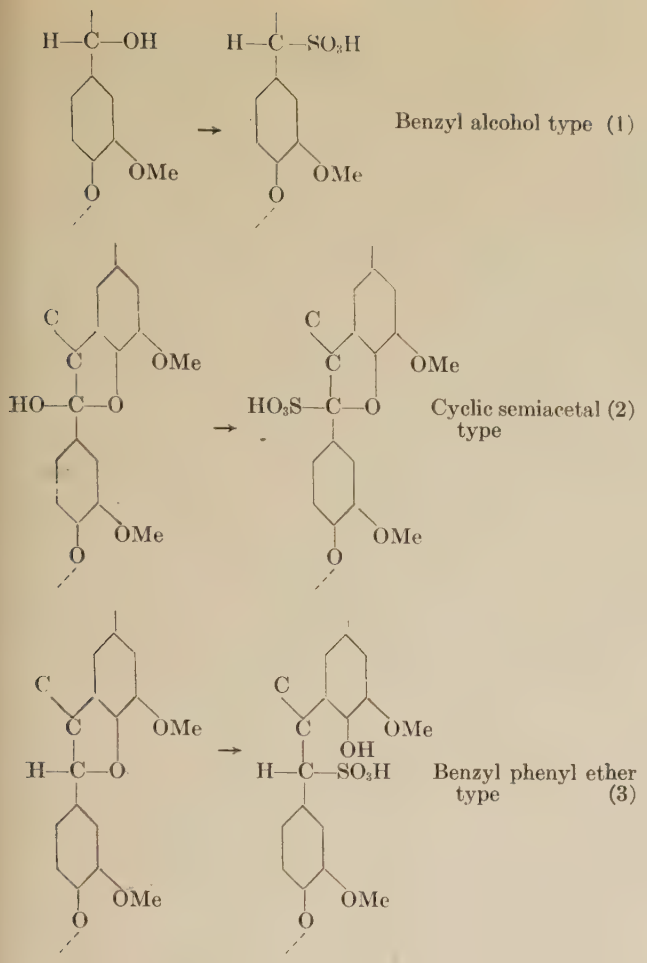
The fact that sulphonation is a relatively mild method of isolating lignin from wood, has been illustrated in a convincing manner by the following result of investigations made by Gunhild Aulin-Erdtman.

She has compared the absorption curves of lignin sulphonic acid with those of other lignin preparations, such as butanol-lignin, acetic acid lignin, the so-called "native lignin" of F. E. Brauns, etc. Among all these the curve of lignin sulphonic acid shows the most developed absorption band at 280 mmμ as well as the lowest unspecific absorption in the long-waved ultraviolet.

It is extremely important that we learn the chemical reaction mechanisms for the sulphonation and dissolution of lignin. This problem is at present the object of great interest and much research is being conducted in this direction in laboratories both in Europe and America.

There are at present three types of reactions that command the most interest, namely: (1) the exchange of alcoholic hydroxyl groups in the benzyl alcohol position for sulphonic acid groups (2) sulphonation of hydroxyl groups in cyclic semiacetals, and (3) sulphitolytic splitting of benzylphenylethers.

Fig. 2. Spruce cross section after reaching 120°C during sulphite cooking



It is obvious that only one type of reaction is insufficient to explain all the phenomena that are observed in connection with the sulphonation of lignin and all properties of the lignin sulphonic acid formed. It would appear more probable that the side chains in different guajacyl-propane units represent several different structures. As yet it has been impossible to set forth a formula that would explain all of the facts as they are now known.

A discussion of the chemistry of the sulphite process would be incomplete if it were limited to the reaction between lignin and sulphite. We have been able to show, by investigations carried on about 20 years ago, that the sulphite, in a very interesting manner, also reacts with sugars that are dissolved from the wood by

Fig. 3. Spruce cross section after reaching 135 °C. during sulphite cooking

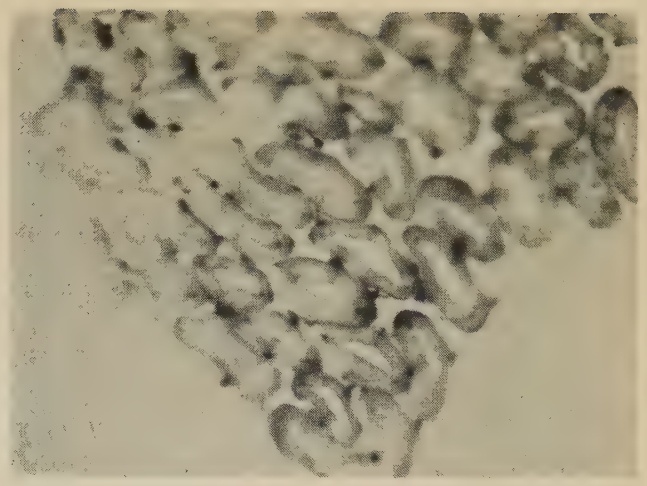


Fig. 4. Spruce cross section after 1 hour sulphite cook at 135 °C.

the action of the acid-cooking liquor. These sugars arise from the easily hydrolyzable wood polyoses and consist of mannose, xylose, some arabinose and galactose and, with strong acid cooking, even some glucose.

The sulphite waste liquor contains, on the other hand, a considerably smaller amount of sugars than would be expected from the amount known to have been dissolved from the wood. The reason for this is that a loss of sugar occurs during the cooking. The degree of this sugar destruction increases with increasing base content in the cooking acid. In this way 40 to 50% of the total amount of dissolved sugars is destroyed in a strong pulp cook, while in a rayon pulp cook only 30 to 40% is destroyed.

It is clear that this loss of sugars is very important when use is made of the waste liquor's sugar content for the fermentative production of alcohol, or other ferment-processes, or for the cultivation of fodder yeast.

The cause of this destruction of sugar has been shown to lie in some very interesting reactions between the aldoses and the bisulphite.

One of these reactions is, surprisingly enough, an oxidation of aldoses to aldonic acids. This reaction is, in reality, an oxido-reduction process where the bisulphite acts as an oxidizing agent for the aldoses while it is itself reduced to thiosulphate. We have formulated this reaction in the following manner:

$$\begin{array}{l} \mathrm{CHO} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} + \mathrm{HSO_3^-} \rightleftarrows \begin{array}{l} \mathrm{SO_3^-} \\ \mathrm{CHOH} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} \qquad (1)$$

$$2 \begin{array}{l} \mathrm{SO_3^-} \\ \mathrm{CHOH} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} + 2\mathrm{HSO_3^-} \rightarrow 2 \begin{array}{l} \mathrm{SO_3^-} \\ \mathrm{C=O} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} + \mathrm{S_2O_3^{--}} + 3\mathrm{H_2O} \qquad (2)$$

$$\begin{array}{l} \mathrm{SO_3^-} \\ \mathrm{C=O} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} + \mathrm{H_2O} \rightarrow \begin{array}{l} \mathrm{O} \\ \mathrm{C-OH} \\ \mathrm{(CHOH)_4} \\ \mathrm{CH_2OH} \end{array} + \mathrm{HSO_3^-} \qquad (3)$$

The first step is the well-known reversible addition of bisulphite to sugar by the formation of an aldehyde-

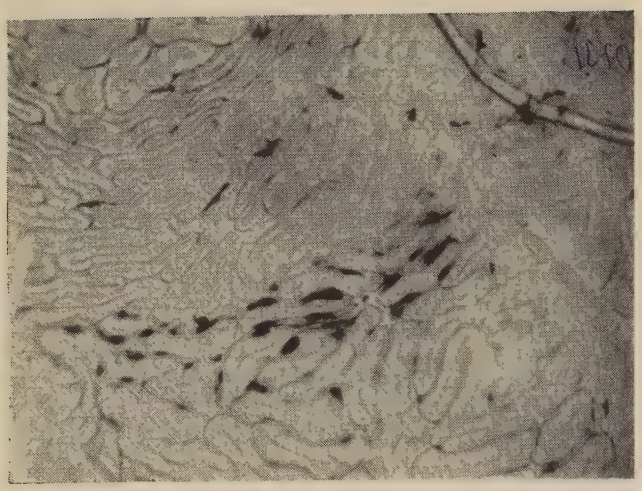


Fig. 5. Spruce cross section after 3 hours sulphite cook at 135 °C.

bisulphite compound. This later undergoes an oxido-reduction with bisulphite resulting in the formation of an α-keto sulphonic acid as the oxidation product and a thiosulphate ion as the reduction product. This intermediate hypothetical keto-sulphonic acid decomposes, apparently immediately, by hydrolysis into the final product, an aldonic acid. This reaction has not only been accomplished with pure sugars but its occurrence in the sulphite process has also been demonstrated in collaboration with T. Johnson by the isolation of mannonic and xylonic acids from sulphite waste liquors.

In addition to this pure oxidation of sugar there occurs also to a small degree a formation of sugar derivatives which contain both carboxyl and stable sulphonic acid groups.

Such sulphonic-carboxylic acids have been obtained in a crystalline form from model reactions with glucose, and their constitution has, in part, been clarified by the work of E. Adler. Their existence in sulphite waste liquor has also been substantiated.

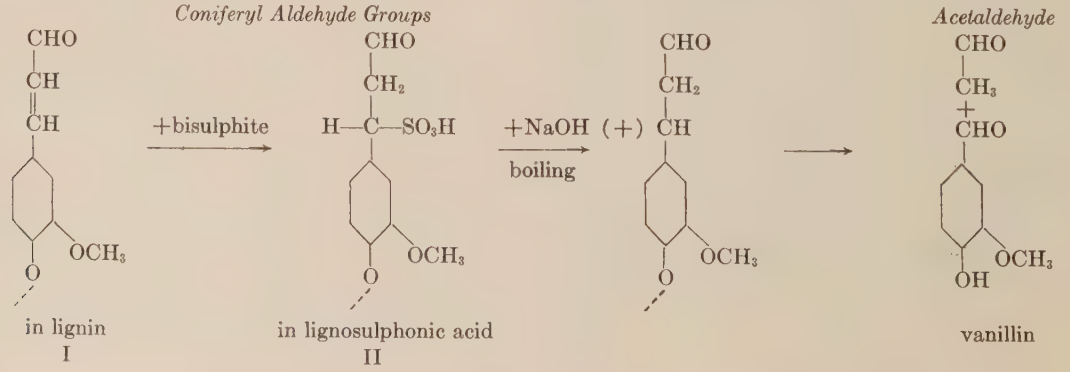
From the maze of reactions that take place during the sulphite cooking, a number of other aldehydic substances in addition to sugars, are formed, and these demand a certain interest in connection with the well-known concept of "loosely combined SO_2." This fraction of the waste liquor's sulphurous acid content is distinguished by the fact that it cannot be titrated directly with iodine but can be liberated instantaneously by alkalizing the liquor at room temperature. This "loosely combined sulphite" can cause undesired effects in two different connections: in the fermentation of the waste liquor's sugars it can effect the functioning of the yeast cells, and in the evaporation and burning of waste liquors, it can, together with the $CaSO_4$ found in the liquor, cause the annoying phenomena of scale in the heating tower of the evaporating apparatus. Adler has therefore studied the chemical nature of the substances in waste liquor which add on bisulphite in this "loosely combined form." It was found that the sugar bisulphite compounds were of little importance in this connection as they are decomposed early in the treatment of the liquor. On the other hand, it was found that the waste liquor contains low molecular, volatile aldehydes, particularly formaldehyde and methyl glyoxal, which form highly stable addition compounds with bisulphite which are carried over into the neutralized liquor. Even the lignin sulphonic acid contains some of this so-called "loosely combined sulphite" in a relatively stable combination.

In lignosulphonic acid, however, only part of the "loosely combined sulphite" is bound to aldehyde groups. As Adler has recently shown, the lignosulphonic acid molecule contains a small percentage of sulphonated coniferyl aldehyde groups (II).

These groups have been formed during the sulphite cook from coniferyl aldehyde units (I) which occur in the native lignin molecules. There is only one such unit for each 40 to 60 methoxyl groups. Nevertheless, these units are responsible for the well-known color reactions of wood, as for example the violet-red color produced by phloroglucinol-hydrochloric acid or the yellow colors produced by aromatic amines. If a solution of lignosulphonic acid is treated with alkali for a few minutes at room temperature, the sulphonic acid group in II is split off and the unsaturated coniferyl aldehyde side chain is regenerated which can easily be demonstrated by addition of phloroglucinol-hydrochloric acid or a similar reagent. By boiling alkali these units are broken down to acetaldehyde and vanillin. Thus, the well-known formation of vanillin by heating sulphite waste liquors or lignosulphonic acids with alkali is, at least partly, explained by this reaction. The amount of coniferyl aldehyde groups present in lignosulphonic acid corresponds, however, only to about one third of the total yield of vanillin obtained. The mechanism of the formation of the residual two thirds of vanillin is still an unsolved problem.

EVAPORATION OF SULPHITE WASTE LIQUOR

The problem of utilizing waste liquors from the sulphite industry is almost as old as the industry itself. One only has to remember that about half of the dry weight of the wood, including most of the lignin and a considerable part of the hemicellulose components, is



lost in the waste liquor and is not utilized for any constructive purpose. In Sweden, a large part of the fermentable sugars are, it is true, utilized by conversion into alcohol. Thus, there are, at present, alcohol plants connected with no less than 34 of the 58 sulphite mills in the country. In 1946, 48 kg. of 100% alcohol were obtained per ton of sulphite pulp which, that year, amounted to 1.2 million tons. During the Second World War, when this production was of the greatest importance for the support of Sweden, and the alcohol was used among other things, as a motor fuel, the yield was increased to 65 kg. per ton of the total production of pulp. The best yield of alcohol obtained in practical operation, is about 75 kg. per ton of unbleached strong pulp and 125 kg. per ton of rayon pulp. During this time the conditions in Sweden, due to the blockade, were such that the alcohol, which formally had been a by-product, almost became the most important product of the sulphite industry. In order to increase the alcohol yield, a special type of pulp was prepared by cooking at a relatively high acidity and temperature. The pulp obtained was used as cattle fodder.

Since the war the price of sulphite alcohol has dropped greatly and it is a question whether or not, in the future, it can compete with synthetic alcohol. That is, under the assumption that the world will return to normal conditions.

Also during the war, the question of evaporation and burning of sulphite waste liquor became important. Even during the First World War for that matter, attempts had been made to solve this problem but without success. Further investigation was carried out during the time between the two wars, but only a few smaller evaporation units were in operation at the outbreak of the war in 1939.

There are two main considerations that have forced the development of a method of evaporation of sulphite waste liquor in Sweden. One is the fuel supply situation in the country, and the other a question of water pollution by waste liquors. One must, when evaluating the possibility of evaporating waste liquors in Sweden, consider the fact that the country is practically void of natural fuels such as coal and oil. During the war, when it was blockaded for several years, it was necessary to tax the forests very severely in the quest for fuel. Large amounts of high grade pulp wood were burned, not only in the boilers of pulp mills, but also to heat large cities such as Stockholm. It is therefore only natural that the attempts to solve technically and economically the problem of using waste liquor as a fuel were intensified during the war. This work is still going on with undiminished vigor. The high price of coal also has a stimulating effect upon this research. Coal costs today in Sweden 75 kronor per ton, that is about 20 dollars. Even if one can count on this price being reduced, the high fuel costs must still be considered. The decreasing price of alcohol may possibly force the closing of some alcohol plants and this in turn cause the respective sulphite mills to adopt the waste liquor evaporation and burning procedure.

From the economic viewpoint it can thus be seen that the ground is well prepared for the adoption of a policy of evaporating waste liquors in Sweden. The interest has therefore been very great, and much work has been done in an attempt to find a technically and financially sound solution. To consider the actual problems regarding the evaporation of waste liquors, a committee consisting of members from both industry and research was formed in 1945 in the Swedish Forest Products Research Laboratory.

The greatest difficulty in attempting to evaporate waste liquor is the scale formation on the heating surfaces of the evaporation apparatus. If these scales are allowed to grow unmolested, the heat transmission is decreased and the pipe lines and heat exchangers are eventually clogged. Therefore, one of the committee's first and most urgent assignments was to examine the conditions of formation of these scales and to attempt to find a way to prevent their formation.

To begin with samples of these scales were obtained from several mills and these were examined by chemical and x-ray means. In this manner it was confirmed that the scales consisted practically completely of calcium sulphate. Calcium sulphate exists in three different modifications: the dihydrate, semihydrate, and the anhydrite. The solubility of these as a function of temperature has been determined by, among others, Partridge and White, as well as by Hill. It is a fundamental characteristic of these salts that the solubility decreases sharply with increasing temperature. It is also possible to determine in which form the calcium sulphate will precipitate during evaporation. At temperatures below 98°C. the dihydrate precipitates, above this temperature the semihydrate. Both of these are converted slowly into the stable anhydrite form. This conversion takes place, however, very slowly at lower temperatures. It was found that apparatus that worked at a temperature under 100°C. formed mainly the dihydrate. At higher temperatures mainly the anhydrite was formed—or sometimes a mixture of semihydrate and anhydrite. It might be pointed out in this connection that it is not only in the evaporation equipment that incrustation occurs and causes difficulties. The alcohol plants too can be affected, especially the distillation columns.

The sulphite waste liquor always contains some sulphate ions in addition to calcium ions which are present in excess. The prerequisite condition for the precipitation of calcium sulphate is therefore always present. The tendency for such a reaction to occur is naturally greater, the greater the concentration of sulphate ions in the liquor. Therefore, it is desirable to hold the concentration of these ions in the liquor as low as possible. On this basis the committee began to examine systematically the sulphite process in order to ascertain in which phase and in what manner the sulphate ions arise.

During the preparation of the cooking acid a larger or smaller amount of sulphate ions is formed.

In order to examine the formation of sulphate ions during a sulphite cook, a number of test cooks were performed in glass tubes to avoid catalytic effects from the walls of the autoclave. The glass tubes were placed in steel tubes for protection and these were rotated in an oil heating bath. At regular intervals the tubes were removed and allowed to cool. The cool liquor was then analyzed. By this method, data obtained for the formation of sulphate during a sulphite cook as a function of cooking time, showed that the sulphate

formation becomes very great toward the end of the cook. This fact was known, in general, from earlier studies.

An entirely different system was shown, on the other hand, by the data from a sulphite cook when the samples were taken directly from the digester in a mill. Instead of increasing, the sulphate content in the liquor continually decreases at the beginning of the cook and has a minimum value some hours before the end of the cook, after which the sulphate content increases again, somewhat, toward the end of the cook.

The obvious contradiction between these later analyses and the experiments in the glass tubes can be explained in a simple manner if one assumes that calcium sulphate was precipitated during the cook. The gypsum was apparently precipitated in a finely divided form on the fibers. In the experiments in the glass autoclaves when the liquor was allowed to cool in contact with the pulp, the gypsum was redissolved, and the analysis showed the total amount of newly formed sulphate. In the mill, on the other hand, the warm liquor was removed from the digester and the analysis showed only the momentary sulphate ion concentration in the liquor. We later reproduced very closely the mill results when the laboratory cooking was accomplished in such a manner that the samples could be taken during the course of the cook.

We have also examined the changes in sulphate content in liquor during its passage through an alcohol plant. The liquor here undergoes three treatments: neutralization, fermentation, and distillation. These processes showed themselves, in the meanwhile, to have little effect on the sulphate ion concentration in the liquor. In general, this concentration was about 800 to 1500 mg. per l. irrespective of the processes of the alcohol plant.

Thus we come to a discussion of the behavior of waste liquor in the multiple effect evaporation equipment. As the liquor gradually thickens during its passage through the apparatus, a greater or lesser amount of the sulphate is precipitated in the form of calcium sulphate, due to the fact that the saturation limit is passed.

The gypsum can precipitate during evaporation in two ways: partly as a finely dispersed suspension and partly as a hard crust on the heat transfer surfaces of the evaporation equipment. Guided by laboratory experiments and experience from useful procedures in actual operation, attempts were made to ascertain which factors determined whether calcium sulphate would precipitate in the one form or the other. One can express the conditions during evaporation by use of a "distribution factor" I. This is defined as the quotient of the amount of calcium sulphate precipitated as scale and the total amount precipitated. Thus

$$I = \frac{\text{amount } CaSO_4 \text{ precipitated as scale}}{\text{total amount of } CaSO_4 \text{ precipitated}}$$

This factor should be as little as possible if the evaporation apparatus is to work without interference. If it is large, care must be taken to see that the incrustation is removed before it becomes too great. This is generally done by washing with water or the acid condensate formed during the evaporation.

Of the factors that effect I, the most important is the temperature difference between the liquor and the heating surfaces. This should be as little as possible.

Another circumstance of great importance is the speed of the liquor over the heating surfaces. The greater this is, the less tendency the calcium sulphate shows toward forming scales.

The distribution factor has also shown itself to be dependent upon the solids content of the liquor. The evaporation of a thick liquor leads to a greater amount of calcium sulphate being precipitated in a finely divided form than the evaporation of a thin liquor does. It is therefore advantageous to conduct the evaporation at as high a concentration of solids as possible in the circulating liquor.

An interesting method that has been attempted with a view toward preventing incrustation on the heating surfaces is a rapid heating of the liquor by means of superheated steam before it is allowed to enter the evaporation equipment. In this manner it is possible to precipitate some of the sulphate in a finely dispersed form and thus maintain the heating surfaces relatively clean.

During our work on the Committee for the Evaporation of Sulphite Waste Liquor we have been lead also to a study of the nature of "loosely combined sulphite" and its changes during the passage of the liquor through the sulphite mill, the alcohol plants, and the evaporation unit. Some interesting results were obtained. In the evaporation of fermented liquors from the alcohol plants also scales of calcium sulphite can sometimes be obtained. The fermented liquors have, in general, a pH of between 4.5 to 6 and during evaporation a large amount of this "loosely combined sulphite" is precipitated. By the evaporation of acid liquor, on the other hand, a liberation of sulphur dioxide and volatile aldehydes occurs. These combine in the condensate to strongly acid α-hydroxy sulphonic acids.

These results, that have just been mentioned, have made it possible to limit the occurrence of scales. It is, however, impossible to prevent it completely. Therefore, arrangements have been made to remove the incrustation that occurs without interrupting the continuity of the procedure.

Without going into too many technical details, the author will briefly describe two evaporation systems that are currently in use in Swedish sulphite mills.

The equipment for the evaporation of acid liquor supplied by AB Rosenblads Patenter operates in three steps. Each step consists of a heat exchanger and a steam separator. The heat exchanger is of a lamina type, that is, it consists of a group of plates between which the heating medium passes. Thus waste liquor and condensed steam are contained between alternate plates. By an ingenious system of valves the liquor and steam can be made to exchange places. In this manner the condensate from the steam can be used to dissolve the incrustation of the plates before they have become too thick. In order to increase the dissolving effect of the condensate, it is allowed to circulate over the heating surfaces with the aid of special pumps. Incrustation can, in this manner, be held within reasonable limits. Generally the liquor and steam are allowed to exchange places every 8 hours.

The temperature of the waste liquor in the three effects are, respectively, 136, 123, and 110°C. The thin liquor from the alcohol factory first enters effect 3. From there it goes to number 1 and finally to number 2.

From this last effect now having a solid content of 55% it is pumped directly into the boiler house and is burned by injection in the same manner as fuel oil.

The entering steam goes first into the heat exchangers in effect 1. The steam leaving the last apparatus is used in the distillation columns of the alcohol plants. It has a temperature of 110°C. Thus, the evaporation procedure is under pressure all the time. It can be seen that this utilization of the steam is particularly advantageous from an economic viewpoint. Actually only the heat corresponding to a temperature drop from 158 to 110°C. is used for the evaporation, since the heat of condensation of the steam can be used for other purposes. However, for a long time there was little inclination to build an apparatus for pressure evaporation due to the essentially greater risk of incrustation that occurs at higher temperatures. Nevertheless the results, so far, have been very encouraging and it is believed that further development will generally be directed toward the use of higher temperatures for the evaporation of sulphite waste liquor.

The apparatus built by AB Raméns Patenter makes use of the aforementioned method of trying to prevent precipitation of scales on the heating surfaces by means of heat treatment of the liquor.

This apparatus works in five stages. Here also, each stage consists of a heat exchanger and an expansion vessel. The alternating of liquor and steam is, however, not used. Instead the heat exchangers can quickly be taken out of use for cleaning. Generally a quarter of an hour washing per eight-hour shift is sufficient.

The liquor enters effect 3 and then passes through the others in the following order: 3–4–5–1–2, after which it is removed to the boiler house and burned by injection.

Between apparatus number 5 and 1, there is a vessel for the heat treatment of the liquor. In this some of the calcium sulphate is precipitated as a slime and in this form can be carried along in the liquor without causing any difficulty. Some of the liquor from the 5th effect is treated in a similar apparatus, and then returned to number 4.

Some of the steam leaving the first effect is removed for use in the alcohol plant while the rest is conducted, in the normal manner, to No. 2. Fresh steam at 6 atmospheres pressure is used for the heat treatment units between effects 4 and 5, and 5 and 1.

We have also conducted research on many other questions dealing with the use of sulphite waste liquors as fuel. One of the most important of these questions is what should be done with the enormous amount of ash that is formed during burning. How important this question is can be better understood when one considers that 150 kg. ash are obtained from every ton of pulp produced. This value corresponds approximately to the consumption of limestone. The density of the ash is, however, so low that it requires a space which is 10 to 20 times greater than that of limestone. These and many other questions in this field occupy our time today and we hope, in the future, to be able to arrive at satisfactory solutions.

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Weight Factors for Cotton Linters

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It has been noted that the weight factor (1.0) applied to rag stock in fiber analyses appears too low when applied to an analysis of papers containing cotton linters. Because of the increased interest on the part of the paper industry in such raw material, it was decided to make weight factor determinations. Both first-cut and second-cut linters were used in the investigation. Since it appeared from previous work (*1*) that the freeness of this type of stock might affect the value of the weight factor, it was also deemed desirable to study pulps at various degrees of freeness.

The present investigation included a sample of unbeaten second-cut cotton linters and 14 samples beaten to various degrees of freeness, ranging from 435 to 840 ml. (Schopper-Riegler) and also a sample of unbeaten first-cut cotton linters and nine samples beaten to various degrees of freeness, ranging from 380 to 845 ml. (Schopper-Riegler). The standard pulp used in admixture with the linters for the determinations was eastern softwood bleached sulphite (745-ml. Schopper-Riegler freeness). With the exception of the unbeaten cotton linters, all samples were in the wet-pressed state with preservative added. The unbeaten linters were in the air-dried condition.

Thin pads were prepared from suspensions of the pulps and allowed to air dry. An equal weight of the sulphite pulp was mixed with each sample of air-dry cotton linters pulp by suspension in water, followed by thorough stirring with a milk-shake electric mixer. Several thin pads of each mixture were formed on a small Buchner funnel fitted with a hardened filter paper.

A suspension was made from each of the pulp mixtures by removing small pie-shaped pieces from the pads made for any one sample. Standard fiber slides (six in a set) were prepared, stained with "C" stain, and the fibers counted.

The counts obtained from the 12 slide ends in each set were totaled. The number of sulphite fibers multiplied by the weight factor for softwood sulphite (0.9) equaled the number of cotton linters fibers multiplied by the weight factor for linters, since equal weights of pulps were used in the mixture. The resulting weight factors are listed in Tables I and II. Because of the relatively large differences found between the value for unbeaten linters and the various beaten samples, additional sets of slides for fiber count were prepared.

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It will be seen in Table I that, with the exception of the unbeaten linters, the two operators did not make their analyses on the same freeness samples. The samples made for operator B above 600-ml. Schopper-

Table I. Weight Factors of Second-Cut Cotton Linters

Sample no.	Approx. Schopper-Riegler freeness, ml.	Weight factors, operator A	B	Av.
		Unbeaten fibers		
I-a	885	1.91	..	..
I-b	...	2.00	..	..
II-a	...	1.92	..	..
II-b	...	1.72	..	..
III-a	...	..	1.94	..
III-b	...	..	1.89	..
				1.90
		Beaten fibers		
1	840	..	1.50	
2	815	1.46	..	
3	785	1.45	..	
4	770	..	1.45	
5	750	1.52	..	
6	740	..	1.39	
7	725	1.36	..	
8	705	..	1.39	
9	620	..	1.27	
10	600	1.29	..	
11	540	..	1.23	
12	510	..	1.10	
13	470	..	1.12	
14	435	..	1.06	

Riegler freeness were attempts to duplicate the pulps used by operator A. The over-all agreement of results is very good, as it is with the unbeaten fibers.

The data listed in Table I are plotted in Fig. 1. The curve illustrates to what extent the weight factor is a function of freeness in the second-cut cotton linters, at least within the freeness range above 400. Hence, if one knows the approximate freeness of the sample, a fairly close weight factor can be used in fiber analysis. It was decided to take a weight factor by averaging values taken at intervals of 25 units from the curve. The value obtained is 1.34, which corresponds to approximately 680-ml. Schopper-Riegler freeness.

The values determined on the first-cut linters are given in Table II. Again, the results of the two operators are listed separately. In fact, because of the paucity of data collected by operator A, it was thought desirable

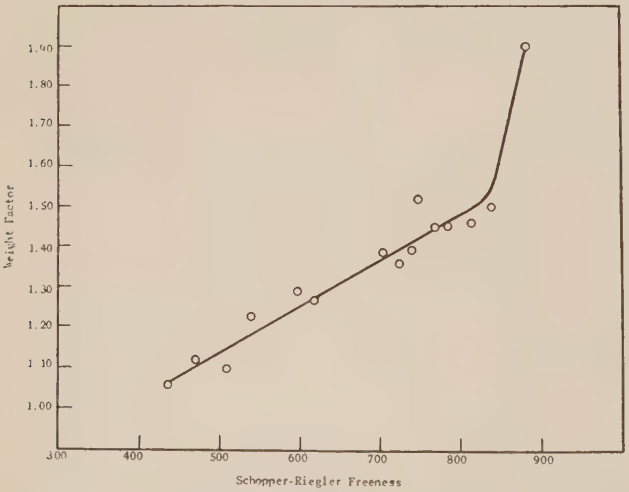

Fig. 1. Weight factors vs. freeness for second-cut cotton linters

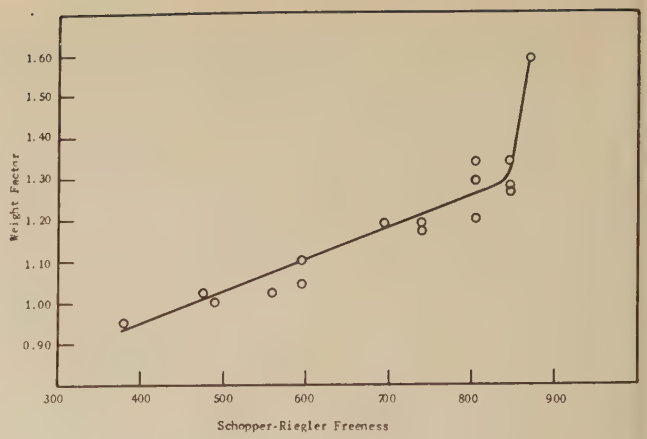

Fig. 2. Weight factors vs. freeness for first-cut cotton linters

to have operator B repeat some of the determinations; hence, under B, columns headed 1 and 2 indicate separate trials. The curve in Fig. 2 shows that, for the first-cut linters also, the weight factor is a function of freeness. The weight factor obtained from this curve by averaging values taken at intervals of 25 units is 1.13. This corresponds to approximately 635-ml. Schopper-Riegler freeness.

Table II. Weight Factors of First-Cut Cotton Linters

Sample No.	Approx. Schopper-Riegler, freeness, ml.	Weight factors, operator A	B 1	B 2	Av.
		Unbeaten fibers			
I-a	870	..	1.31	..	..
I-b	...	..	1.76	..	..
I-c	...	..	1.72	..	..
I-d	...	1.56	..	..	..
II-a	...	..	1.72	..	..
II-b	...	1.47	..	..	..
					1.59
		Beaten fibers			
1	845	1.26	1.34	1.28	1.29
2	805	1.29	1.34	1.20	1.28
3	740	..	1.19	1.17	1.18
4	695	..	1.19	1.19	1.19
5	595	..	1.10	1.04	1.07
6	560	..	1.02	..	1.02
7	490	..	1.00	..	1.00
8	475	..	1.02	..	1.02
9	380	..	0.95	..	0.95

It is evident from a comparison of the curves of Figs. 1 and 2 that not only is the weight factor at the same freeness lower in the case of first-cut linters, but the slope of the curve in Fig. 2 is more gradual than in Fig. 1.

The weight factors found for the cotton linters is appreciably higher than the value (1.0) now used for rag stock except when the linters have been beaten to a freeness in the neighborhood of 400-ml. Schopper-Riegler. Thus, the source of the cotton fiber is apparently of considerable importance in addition to the observed effect of freeness. What can be done is not so obvious. If one knows the exact nature of the furnish, these factors can be taken into consideration; it is the unknown which will cause difficulty.

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1. Graff, J. H., *Paper Trade J.* 119, no. 15: 41–42 (Oct. 12, 1944); *Tech. Assoc. Papers* 27: 471–472 (1944).

Received April 25, 1950.

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Hugn, M. Banfill, Chemist, Howard Smith Paper Mills Ltd., Cornwall, Ont., Canada, a 1946 graduate of Bishop's University.

Harold E. Cox, Chief Design Engineer, The Wallace Press, Inc., Chicago, Ill. Attended the Armour Institute of Technology.

Nils Falk, Engineer, Gavle, Sweden, a 1943 graduate of State College.

Hugo Gaegauf, Manager of Technical Section, Paper Mill on Sihl, Zurich, Switz., a 1929 graduate of Federal Technical Highschool, Zurich.

Nelson F. Getchell, Assistant Manager, Fiber Dept., Ludlow Mfg. & Sales Co., Boston, Mass., a 1938 graduate of Lowell Textile Institute.

Edward C. Hale, Chemical Engineer, Abitibi Power & Paper Co. Ltd., Central Research and Development Div., Sault Ste. Marie, Ont., Canada, a 1948 graduate of McGill University.

Frederick P. Heil, Chemist, Lockport Felt Co., Newfane, N. Y., a 1939 graduate of New York State College of Forestry.

Max W. Heiniger, Mill Superintendent, Papierfabrik Cham A. G., Cham, Switz., a 1944 graduate of the University of Darmstadt.

John T. Henderson, Trainee, Rayonier, Inc., Hoquiam, Wash., a 1950 graduate of Pacific Lutheran College.

Atsushi Inoue, President, Tomoegawa Paper Mfg. Co. Ltd., Tokyo, Japan, a 1921 graduate of Tokyo University.

Raymond C. Koehn, Assistant to Chief Chemist, Packaging Materials Control, General Mills, Inc., Minneapolis, Minn., a 1936 graduate of St. Mary's College. Attended the University of Minnesota Graduate School, 1936–38.

Richard A. Krauss, General Superintendent, Papierfabrik Wilhelmstal, Krebsoege, North-Rhine, Trizone, Germany.

William H. Kreiling, Chemist, Lowe Paper Co., Ridgefield, N. J., a 1949 graduate of Brooklyn Polytechnic Institute.

William F. Llewellyn, Project Engineer, Marathon Corp., Menominee, Mich., a 1941 graduate of Rutgers University.

Frank W. Lorey, Student, New York State College of Forestry, Syracuse, N. Y.

William D. MacKinnon, General Superintendent, Insulation Div., Donnacona Paper Co. Ltd., Donnacona, P. Q., Canada, a 1925 graduate of Queens University.

Charles R. G. Maynard, Research Engineer, Wiggins Teape Group Research Organization, Aberdeenshire, Scot., a 1946 graduate of Pembroke College, Cambridge.

Hisashi Nakamura, Managing Director, Hokuetsu Paper Mfg. Co. Ltd., Tokyo, Japan, a 1907 graduate of Kyoto University.

Turuo Okada, Chief Engineer, Hokuetu Paper Mfg. Co. Ltd., Niigata, Japan, a 1933 graduate of Kyoto University.

Byron L. Pettiss, Quality Control Chemist, International Paper Co., Mobile, Ala.

Marvin C. Raether, Laboratory Technician, Flambeau Paper Co., Park Falls, Wis., a 1949 graduate of the University of Wisconsin.

Thomas F. Ryan, Chemist, Doeskin Products, Inc., Mt. Tom, Mass. Attended Rensselaer Polytechnic Institute.

Ryo Shinoda, Chief Engineer, Tomoegawa Paper Mfg. Co. Ltd., Mochimune Shizuoka City, Shizuoka Pref., Japan, a 1921 graduate of Tokyo University.

Edward H. Snider, Junior Chemist, Canadian International Paper Co., Gatineau, P. Q., Canada, a 1949 graduate of the University of Toronto.

Edward Strazdins, Chemist, American Cyanamid Co., Stamford, Conn., a 1949 graduate of the Technical University, Darmstadt.

Louis M. Stuebe, Trainee, Engineering Dept., Beloit Iron Works, Beloit, Wis., a 1949 graduate of the University of Illinois.

Masao Tashirogi, Director and Technical Superintendent, Nitto Paper Mfg. Co. Ltd., Yamaguchi-ken, Japan, a 1924 graduate of Tokyo Imperial University.

Bernard J. Twisdale, Paper Mill Chemist, Gould Paper Co., Lyons Falls, N. Y., a 1949 graduate of Brigham Young University.

Ralph A. Ulveling, Librarian, Detroit Public Library, Detroit, Mich.

Paul H. Vonderhorst, Assistant Foreman, National Folding Box Co., Inc., New Haven, Conn., a 1939 graduate of Hofstra College.

Averill J. Wiley, Technical Director, Sulphite Pulp Mfrs. Research League, Inc., Appleton, Wis., a 1936 graduate of the State College of Washington, with an M.S. degree in 1937. Attended the University of Wisconsin, predoctoral studies.

James H. Wing, Assistant Research Physicist, Hollingsworth & Whiteny Co., Winslow, Me., a 1949 graduate of Colby College.

Wilfred S. Young, Jr., Editor, American Paper Converter, Howard Publishing Co., Chicago, Ill., a 1937 graduate of Beloit College.

TAPPI Notes

The TAPPI 1951 Year Book is now in preparation. Members of the Technical Association are requested to check their listings in the TAPPI 1950 Year Book and advise the Secretary of TAPPI, 122 East 42nd Street, New York 17, N. Y., of any corrections that should be made. Please be sure that your present position is indicated.

Nils Anderson, Jr., formerly with the Borden Co., is now President of Debevoise-Anderson Co., 155 E. 44th St., New York, N. Y.

Arthur Aronoff, formerly of the New York State College of Forestry is now Chemist for the Ashland Paper Mills.

Lester J. Beaulieu, formerly of the Central Paper Co., is now Manager, Laminating Division, American Coating Mills Division, Owens-Illinois Glass Co., Elkhart, Ind.

Willard E. Carlson, formerly of the Institute of Paper Chemistry is now Chief Chemist, Whiting-Plover Paper Co., Stevens Point, Wis.

Charles H. Chapman of the Hercules Powder Co., has been transferred from Portland, Ore., to the Paper Mill Technical Sales Department in San Francisco, Calif.

Claude Christiansen, formerly of the Weyerhaeuser Timber Co., is now Pulp Mill Superintendent, Potlach Forests, Inc., Lewiston, Idaho.

Ellsworth J. Cook, formerly of the Shawano Paper Mills is now in charge of quality control, Stein Sales Co., Utica, N. Y.

Fred B. Doherty is now Resident Manager of the Ferandina, Fla., Division of Rayonier, Inc.

R. F. Erickson, Vice-President of Rayonier, Inc., has transferred from Fernandina, Fla., to New York, N. Y.

Peter Faichney, formerly of the North Inland Paper Co., is now Asst. Lecturer in Paper Manufacture, College of Technology, Manchester, England.

Loren V. Forman, formerly of the staff of the Institute of Paper Chemistry is now with the Scott Paper Co., Chester, Pa.

James R. Goodearle of the Technical Dept., Pollock Paper Corp., has been transferred from Dallas, Texas to Middletown, Ohio.

Ronald G. Goodwin is now Chief Chemist and Research Manager, Paper & Industrial Appliances, Inc., New York, N. Y.

R. T. Greep is now Production Manager, S. D. Warren Co., Cumberland Mills, Me.

Eric T. Guest, formerly of Bloedel, Stewart & Welsh is now with H. R. MacMillan, Pulp Div., Harmac, B. C.

Sidney Hausman, Consulting Engineer have moved from Westminster, Md., to 340 North Ave., East Cranford, N. J.

Merle A. Heath, formerly of the staff of the Institute of Paper Chemistry is now Associate Director of Development, Chemical Pulp Div., Buckeye Cotton Oil Co., Memphis, Tenn.

D. A. Huber, formerly of the Crossett Paper Mills is now Owner of Huber Feltex Co., 1221 Avon Ave., S. W., Atlanta, Ga.

Arthur P. Hunneman, Consulting Engineer has moved from Metuchen, N. Y., to 116 Cherry St., Elizabeth, N. J.

Norwood H. Keeney, Jr., formerly of the University of Maine is now Paper Chemist for the Fram Corp., Providence, R. I.

H. Hanson Lagerpusch, formerly of the Minnesota & Ontario Paper Co., is now Engineering Chemist in the Technical Sales Dept., Penick & Ford, Ltd., Inc., Cedar Rapids, Iowa.

Robert L. Leaf, Jr., formerly of the Ohio Boxboard Co., is now Technical Director, Shawano Paper Mills, Shawano, Wis.

Robert W. Lull of the American Cyanamid Co., has transferred from New York, N. Y., to Portland, Ore.

Robert F. McDonnell, formerly of Tileston & Hollingsworth is now Mill Manager, Erving Paper Mills, Erving, Mass.

Alfred H. Montville, formerly of the Ecusta Paper Co., is now Manager of the Sidney Hardware Co., Inc., Sidney, Ohio.

Robert H. Mosher, formerly of the Holyoke Card & Paper Co., is now with the Miami Valley Coated Paper Co., Franklin, Ohio. Mr. Mosher is in charge of manufacturing operations and new products development.

H. J. Ostrowski, formerly of the Credit Valley Lime Co., is now with the Brown Corp., La Tuque, P. Q.

Ralph O. Phillips, formerly of the Chemwood Corp., is now Consulting Chemist and Chemical Engineer, Christmas Tree Farm, Londonberry, Vt.

Ralph W. B. Reade, Consulting Engineer for the Ruberoid Co., has transferred from Mobile, Ala., to Joliet, Ill.

John E. Robison, formerly of the Fir-Tex Insulating Board Co., is now Technical Representative for the Kelco Co., Warren, Ore.

Fred W. Sanders, formerly of Pillsbury Mills, Inc., is now with the Eastern Corp., So. Brewer, Me.

Guy A. Schrag, formerly of the staff of the New York State College of Forestry, is now Paper and Packaging Unit Supervisor, Philadelphia Quartermaster Depot, Philadelphia, Pa.

Robert G. Shirriff, formerly of the Coosa River Newsprint Co., is now with the Long Lac Pulp and Paper Co., Terrace Bay, Ont.

James A. Smith, formerly of the Nash Engineering Co., is now Vice-President and Engineer for the Rotareaed Corp., 81 Pondfield Road, Bronxville, N. Y.

John H. Symes, formerly with Excello Paper Products Co., Inc., is now Chemist for Piedmont Paper Products, Inc., Buena Vista, Va.

Walter L. Vignes is now Production Manager of the Calcasieu Paper Co., Elizabeth, La.

Harold R. Wheeler, Jr., formerly of the Rochester Paper Co., is now Paper Technologist, Technical Sales Service Dept., Clinton Foods, Inc., Clinton, Iowa.

A. F. Wendler is now Manager, Specialty Film Sales, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

N. R. Yorke is now Chief Engineer of the new Panelyte Division plant of the St. Regis Paper Co., Kalamazoo, Mich.

* * *

T. C. Duval, Manager of the Development Dept., has succeeded *C. C. Heritage* as the official corporate representative of the Wood Conversion Co., Cloquet, Minn., in TAPPI.

James C. Morris, Production Manager, has succeeded *C. E. Carpenter,* Vice-President (Retired) as the official corporate representative of the Ohio Boxboard Co,. Rittman, Ohio, in TAPPI.

Robert M. McIlvain, Vice-President has succeeded *J. Shartle Brookover* as the official corporate representative of the Downingtown Paper Co., Downingtown, Pa., in TAPPI.

* * *

John A. Minser of the Alloy Sales Section has succeeded *H. J. Bartlett* as the official representative of Crane Co., 836 So. Michigan Ave., Chicago 5, Ill., in TAPPI.

* * *

Recent foreign visitors to the United States have been:

Tore H. Nevrell, Technical Manager, A/B Billingsfors-Langed och Gustafsfors Fabrikers, Billingsfors, Sweden.

Bengt Halling, Mill Manager, Hofrestrom Pulp & Paper Co., Haverud, Sweden.

Sten Th:son Lundberg, Managing Director, Brusafors-Hallefors och Svenskt Pappers A/B, Silverdalen, Sweden.

Sakae Fukuyama, Executive Director in Charge of Engineering and Research, Honshu Paper Manufacturing Co., Ltd., Tokyo, Japan.

Industry Notes

Robert H. Steiner has been appointed Research Coordinator for the Atlas Mineral Products Co., Mertztown, Pa. He has been with the Firestone Tire & Rubber Co., for the past seven years.

* * *

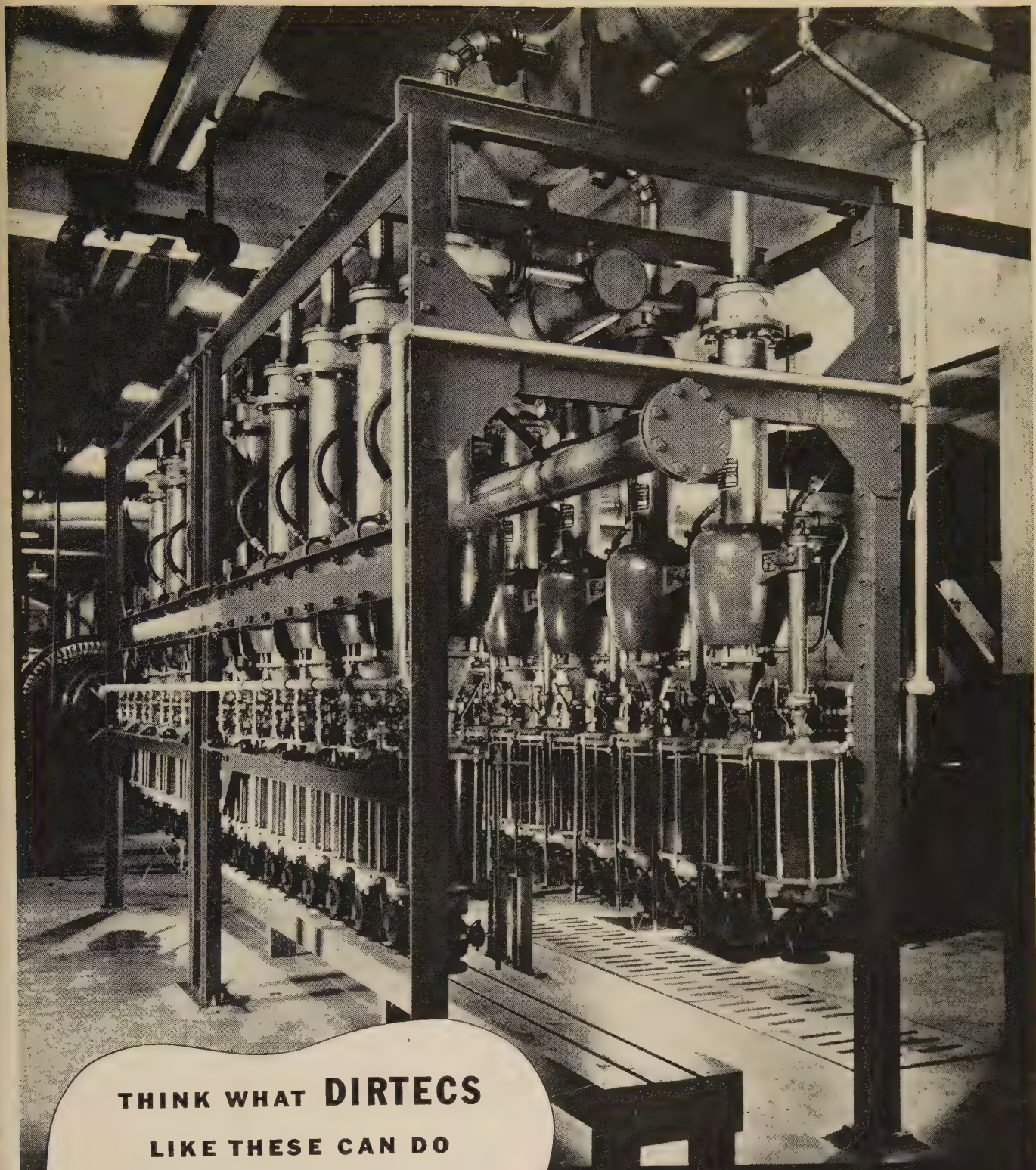
The Babcock & Wilcox Tube Co., Beaver Falls, Pa., has issued a handbook on methods of working seamless tubes and pipe of the intermediate Croloy tubing steels. (Technical Bull. No. 9B).

* * *

A. E. Staley Mfg. Co., have added the following to its industrial sales staff for starches. William A. Gray, formerly of the General Mills Co., has joined the Chicago organization and George A. T. Moore, formerly of the C. E. Chappel Co., Syracuse, N. Y., will be located at Minneapolis. Plans for the construction of a 10,000 bushel soybean processing plant for Painesville, Ohio has been announced. The new plant will use the extraction process. All major equipment has been purchased. Soybeans are currently the nation's largest single source of vegetable oil.

* * *

Otto W. Hein, Production for Shartle Bros. Machine Co., has returned from the Black-Clawson International plant at Newport, England. The foundry at Newport is equipped to



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produce castings of iron, steel, stainless, brass, and aluminum. The plant makes Black-Clawson-Dilts equipment.

* * *

Circular C 478 Colorimetry, a 56-page publication may be obtained from the Publications Section, National Bureau of Standards, Washington, D. C., at 30 cents per copy.

* * *

The Armstrong Machine Works, Three Rivers, Mich., has issued a 2-page bulletin (Bull. No. 206) on ball-float traps for venting air from liquids under pressure.

* * *

The Ingersoll-Rand Co., 11 Broadway, New York 4, N. Y., has issued a new bulletin describing their T-Series stationary air compressors.

* * *

Selected readers of *TAPPI* have received or will receive, from the A. N. Marquis Co., 210 E. Ohio St., Chicago, Ill., requests for information pertaining to their personal specialties within their occupational fields. Information submitted will be reviewed to determine suitability for inclusion in the company's new reference book, Who Knows—And What, 2nd Ed. Data are published of only those persons having skills or knowledge of highly specialized subjects. General professional eminence is not a deciding factor in the selections. This book is a companion book to Who's Who in America and will be of the greatest value when it contains every possible reference. If you or any of your associates should be listed please write to the publisher.

* * *

The Permutit Co., 330 W. 42nd Street, New York 18, N. Y., has acquired from the American Cyanamid Co., its ion exchange business and the trademark "Ionac." These materials will be manufactured and distributed by Permutit's subsidiary, The American Zeolite Corp.

* * *

Poly-Lok, a new adhesive for fabricating multiwall and specialty bags from polyethylene-coated kraft stocks, has resulted in successful packaging for more than a score of products once bothersome or even impossible to handle, according to the National Starch Products Co., 270 Madison Ave., New York 16, N. Y. Poly-Lok is a ready to use cold Resyn seam adhesive that combines high solids with low viscosity. These seams are said to withstand hot calcium oxide at 250°F. and food locker temperatures at −20°F.

* * *

Charles E. Smart has been elected President of W. & L. E. Gurley, Troy, N. Y., 105 year old manufacturers of engineering instruments, including testing instruments used in the paper industry. Mr. Smart has been with the Company since 1920.

* * *

Under the Naval Stores Act, a new Federal standard for tall oil rosin becomes effective on November 1. The standard is defined as follows: "Tall oil rosin means rosin remaining after the removal of the fatty acids from tall oil by fractional distillation. Such rosin shall have the characteristic form and appearance and other physical and chemical properties normal for other kinds of rosin."

Since tall oil rosin is made by distilling off the fatty acids from tall oil, the remaining rosin usually contains a small percentage of fatty acid. It was decided by the U.S.D.A. Production and Marketing Administration not to place a limit on the fatty acid content since this would be controlled as to quantity and kept low enough if the rosin conformed with the prescribed physical and chemical properties which were accepted for the other kinds of rosin.

* * *

The paper industry output has exceeded 99% of theoretical capacity for every week in 1950 except three, and has been over 100% of theoretical capacity for 25 of those weeks. Paperboard has been close behind.

* * *

The nations first industrial research laboratory, the General Electric Research Laboratory, will celebrate its 50th anniversary during the week of October 9. Dr. Willis R. Whitney, a young chemist at Mass. Inst. of Technology was selected to organize and direct the new laboratory which was started in a barn behind the home of Dr. Charles P. Steinmetz.

* * *

At present rates of production, the output of primary paper and board during 1950 will reach an all time high of more than 23 million tons. This estimated output is more than 15% greater than last year's total production, and more than 5% above the previous peak of output of 1948.

Defense spending brought about by the Korean situation is expected to add about 4% to the gross national product.

* * *

The Du Pont Company plans a substantial increase in capacity of its Louisville Works to make neoprene. The plant has a present rated capacity of 60,000 long tons of neoprene a year.

* * *

This year marks the 50th anniversary of the Niagara Alkali Co. It was organized in 1900 under the name of the Roberts Chemical Co. A notable contribution of the company has been to public health by the chlorination of water supplies. On Nov. 22, 1912, it tried out at Niagara Falls the first successful apparatus for this purpose. Niagara Falls then had one of the highest death rates from typhoid of any city in the United States. Two years later the death rate from this cause was nil.

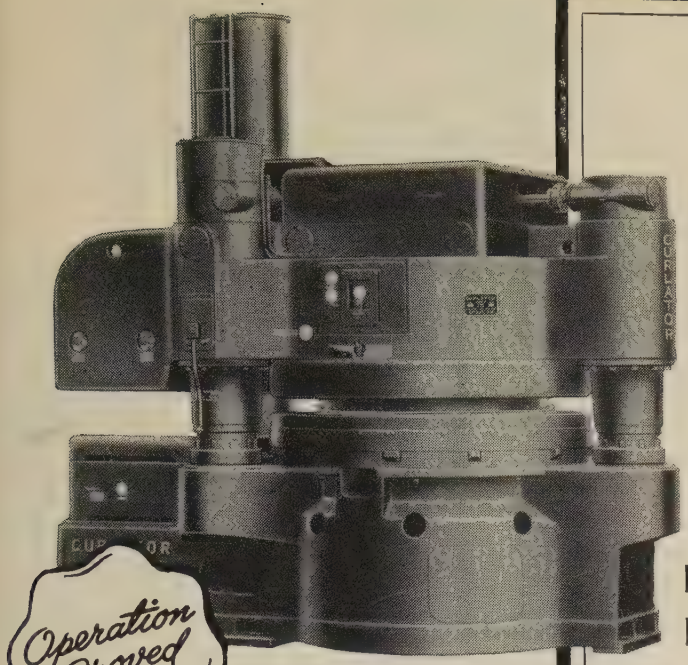
Niagara started with the ideas and some 30 patents of Isaiah L. Roberts, principal of which was an electrotype cell diaphragm to withstand the destructiveness of basic chemical compounds in making caustic potash and bleaching powder. The company has a number of firsts to its credit in this country. Principal of these is the successful introduction of liquid chlorine for bleaching cotton textiles. Former bleaching methods weakened the cloth. The company was the first to manufacture synthetic caustic potash. The flaking of alkalies was introduced just prior to World War I. Prior to then they were sold only in fused, broken and liquid forms. It was the first to supply hydrogen from electrolytic cells, for use in making ammonia. The principal officers of the Niagara Alkali Co., are J. Clarke Cassidy, President, S. Willard Jacobs, and Stephen J. White, Vice-Presidents and William J. Weed, Manager of Sales.

* * *

$1,297,000 has been authorized by the ECA for the purchase of modern machinery in the United States for a sulphate pulp and paper mill at Frantshack, Austria. This mill owned by the Natronzellstoff-und Papierfabrik A/G is Austria's sole producer of sulphate pulp and paper. The plant will have a capacity of 24,000 metric tons a year. The new paper machine will be used exclusively to make multiwall bags for European needs.

* * *

Tests run at the Nekoosa Edwards Paper Co., in cooperation with the Sulphite Pulp Manufacturers Research League showed that, although sulphite liquor can be used as fuel, the cost of using it may be prohibitively high. The principal problem is the amount of water that needs to be evaporated under present conditions.



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This is not a Paul Bunyan story. It has been accomplished and is being done today in actual mill operation. For example last year, by curlating pulp, one newsprint mill saved over 6,500 cords of pulpwood—more than enough to pay for several Curlators. In addition to this outstanding record more than enough sulphur, coal and limestone was saved to pay all this Curlator's operating and maintenance costs.

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James A. Creswell has been appointed purchasing agent by the Spruce Falls Power and Paper Co., Long Lac Pulp and Paper Co., Ltd., Kimberly-Clark Corp., of Canada Ltd., and the Upper Canada Timber Co., Ltd. J. D. Hoffman who has been Acting Purchasing Agent has returned to Neenah, Wis., as Asst. General Purchasing Agent for the Kimberly-Clark Corp.

* * *

The following are the official guides for doing business with the Armed Services: "Follow Me, A Guide for Selling to the U. S. Air Forces" (From Commanding General, Air Material Command, Wright Field, Dayton, Ohio). "Selling to the Navy" (From the Government Printing Office, Washington 25, D. C.—15 cents). "Purchasing Items and Purchasing Locations" (From Army Procurement Information Center, Pentagon Bldg., Washington, D. C.). Arrange with the proper procurement office (55 listed in the above booklets with type of products handled) to be notified when your type of product is to be purchased. This can be done by mail. To do classified (secret) production, first arrange with the Defense Department to clear your employees for security, and then arrange to be invited to bid, or negotiate for such work.

* * *

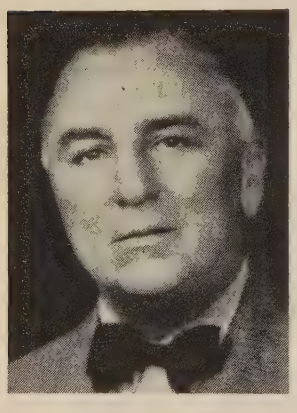
The Folding Paper Box Association of America, P. O. Box 1090, Chicago 90, Ill., has issued a book "The Folding Carton." This is designed to replace the Dept. of Commerce publication "Folding Paper Boxes." Illustrated material includes detailed information for the technical worker.

* * *

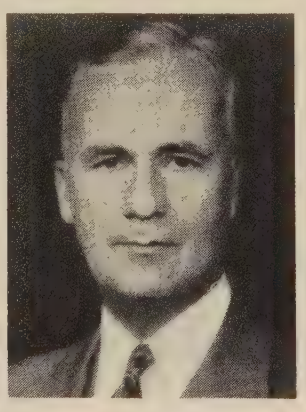
The Pennsylvania Salt Manufacturing Co., celebrated its 100th Anniversary at the Whitemarsh Research Laboratories, Chestnut Hill, Pa., on September 27.

* * *

This is the 25th Anniversary of the Buffalo Electro-Chemical Co., one of the first manufacturers in the country to make hydrogen peroxide electrolytically.



C. A. Buerk



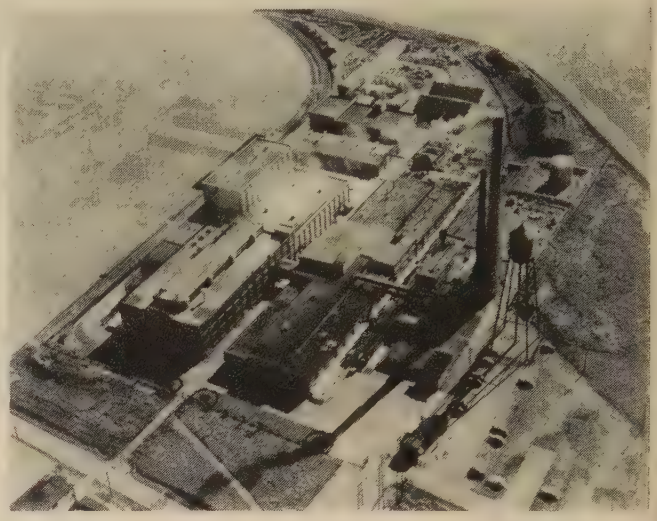
M. E. Bretschger

Mr. C. A. Buerk, who had been in the hydrogen peroxide field since its earliest days in 1907, founded the company. He has been President since 1925. Dr. M. E. Bretschger, Vice-President, joined the organization soon after and has ever since been responsible for its technical development, with Dr. H. O. Kauffmann in charge of research.

Becco Sales Corp., a wholly owned subsidiary acting as sales agent for the company was organized in 1930 to qualify for business in all states.

Today's chief markets for hydrogen peroxide are in the bleaching of textiles, pulp and wood. Over 75% of all cotton bleached in this country today is treated with hydrogen peroxide.

Becco's new Cold Steep bleaching process offers the small operator the efficiency of high densities without post neutralization or use of the pulp mixing, retention or reduction equipment. In this process a pulp sheet is impregnated with a hydrogen peroxide solution of controlled alkalinity. The impregnated pulp in rolls or sheets stands at room temperature for several days and then is added directly to the paper furnish.



Present-day view of the Buffalo Electro-Chemical Co. plant

For bleaching groundwood it is used with sodium silicate. Other alkalies such as trisodium phosphate, pyrophosphates, or caustic soda are used on semichemical or chemical pulps.

In the bleaching operation the pulp sheet is formed either on a Kamyr or Rogers or other feltless wet machine, on a vacuum drum thickener equipped with press rolls so that the sheet is self supporting and at a density preferably between 35 and 50%.

Bleach liquor (about 0.35 gallons per face foot) passes through distributor pipes and to applicator rolls on which brushes assist in spreading it evenly over one or both faces of the pulp sheet. The continuous impregnated web is wound into rolls, or slit and cut in sheets. The pulp is then stored for 1 to 7 days at the mill or in transit. After storage, the sheets or rolls are added to the furnish in the hydrapulper or beater where only slight final pH adjustment of the furnish is required.

Minor changes in bleach liquor formulas adapt the process for bleaching sulphites and semichemical pulps or for the super-bleaching of bleached sulphites and krafts. Adaptation of the Cold Steep Bleaching process to other types of high density equipment is in the active mill development stage.

* * *

The Plaskon Division, Libbey-Owens-Ford Glass Co., is now marketing a modified urea formulation wet strength resin which it claims combined the economy of the ureas with the high strength results obtained with melamines. The Plaskon aminoplast gives high wet strength without the use of alum. It is preferably added to the headbox of the paper machine rather than to the beater.

* * *

A committee of the Manufacturing Chemists' Association is now working on a program to develop fiber drums for pastes, semisolids and liquids. It is expected that the fiber drum can be used to ship many commodities heretofore shipped in metal containers.

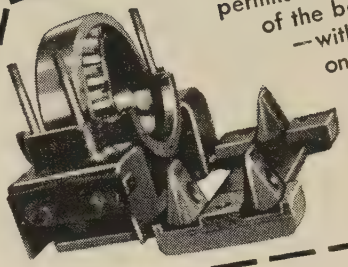
TRAIL BLAZERS to more efficient paper production

Four important developments in paper-making efficiency—some of which are becoming standard practice with bearing manufacturers, *originated with* **SKF**.

THE FIRST

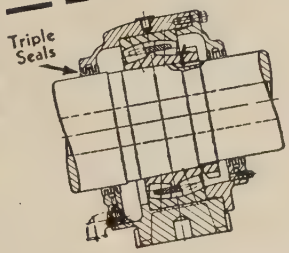
Spherical Roller Bearings applied to paper machinery, with results of smoother paths for power and real operating economies, were SKF Bearings.

THE FIRST



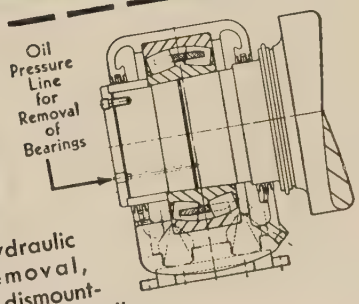
Rocker Mount Design, which permits expansion and contraction of the bearings and driers —with no lateral stresses on the side frames, was an SKF development.

THE FIRST



Triple Seals, for efficient non-rubbing sealing that retains lubricant independent of speed and temperature, were designed by SKF.

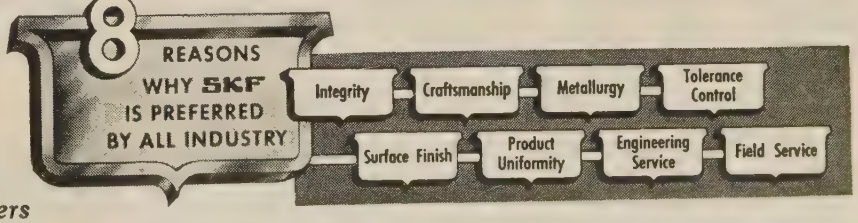
THE FIRST



System for Hydraulic Bearing Removal, where such dismounting of large or small bearings becomes a rapid and easy operation that eliminates damage to bearing and housings, was originated by SKF Engineers.

SKF

BALL AND ROLLER BEARINGS



Yes, SKF, in the paper industry as well as many others, has blazed the trail to more efficient, economical, reliable production. And right now, SKF Engineers in cooperation with machinery builders are *anticipating* the needs of advanced designs of paper machinery — so that years from now production will be even more efficient. **SKF** INDUSTRIES, INC., PHILADELPHIA 32, PA., *the Pioneers of the Deep Groove Ball Bearing—Spherical Roller Bearing—Self-Aligning Ball Bearing.*

7100

Development of the first 100% active flake, nonionic detergent to be commercially produced, was announced recently by Dr. Thomas H. Vaughn, Wyandotte Chemicals Corp., at the Annual Fall Meeting of the American Oil Chemists' Society, San Francisco. This solid surface-active agent is one of an entirely new series of nonionics developed by Wyandotte Chemicals.

According to Dr. Vaughn, vice-president—research and development, "the new nonionic surface-active agents constitute a companion series for the anionic products which Wyandotte has been marketing for years. These new detergent products, commercially named Pluronics, will be offered for many special applications, such as wool scouring, dishwashing, home and commercial laundry operations, and certain metal cleaning processes, where the nonionics have proved to be more satisfactory than the anionics. Particularly effective in hard water areas, it is expected the Pluronics will increase the efficiency of operations in commercial laundries and textile mills."

Until now, nonionic detergents have only been offered in the form of liquids, pastes, or waxlike solids, which severely limited their application. By using a hydrophobic unit not previously used in detergent manufacture, Wyandotte Chemicals' laboratories were able for the first time to produce a solid nonionic. Based on this new unit, a wide variety of 100% active nonionics may be made, ranging in physical form from liquids to a solid of sufficiently high melting point to be flaked on commercial equipment.

The Pluronics have remarkable flexibility—the physical form and surface-active properties may be varied to provide the user with the surface-active agent most suited to his specific requirements. Eventually, the series will include as many products as may be required to cover adequately all of the major fields of application. Pluronic F68, the dust-free flake, will find additional use wherever a 100% active nonionic detergent would be the best type of surface-active agent to use; where the liquid or paste physical form is not desirable.

OBITUARIES

George Harold Young

George Harold Young, Vice-President and Chief Engineer of the Midwest-Fulton Machine Co., Dayton, Ohio, died on September 5 at the good Samaritan Hospital, Dayton, Ohio as the result of an operation.

Mr. Young was born in Lebanon, Ohio on July 7, 1902 and studied mechanical engineering at the University of Cincinnati. He joined the Fulton Engineering Co., Middletown, Ohio in 1922 as a draftsman and eventually became chief engineer, the position he held after the company merged with the Midwest Machine Co., to form the Midwest-Fulton Machine Co., in Dayton, Ohio. In 1945 he was elected a Director and Vice-President of the Company.

He became Secretary of the TAPPI Drying and Ventilating Committee last October and was extremely interested in the progress of this committee although prevented by illness to give the time to it that it was his desire to give.

He is survived by his widow and one son.

Thomas M. Gillespie

Thomas M. "Tommy" Gillespie died in Lake Villa, Ill., on August 29. He was born in Canada on August 20, 1900. Being a Canadian he was a hockey player and starred on the New York Knickerbockers and the Pittsburgh National League teams.

He was a specialist on paper mill supplies and equipment and represented the Lockport Felt Co., Cheney Bigelow Wire Works, and Wyandotte Chemicals, Inc., in the Middle West.

He is survived by his wife, Edna Gillespie.

C. B. Smith

C. B. "Baldy" Smith died on September 6. Baldy, as he liked to be known was an extremely popular salesman in the Middle West, making his headquarters at Kalamazoo. He will long be remembered by his friends in the Kalamazoo Valley for his famous Chincoteague oyster parties at the Gull Lake Country Club.

He was born in Lock Haven, Pa., on July 2, 1886 and started in the paper industry with the New York & Pennsylvania Co., at Johnsonburg, Pa., where he became production manager. After nine years at Johnsonburg he spent two years with the Mattagami Pulp and Paper Co., in Canada as asst. general manager, two years with the Bogalusa Paper Co., as general superintendent, and four years with the American Writing Paper Co., as boss machine tender.

He then entered sales work with the Dyes Distributors Corp., in Chicago as manager of paper sales where he remained for six years. He then joined the Huron Milling Co., at Harbor Beach, Mich., as a sales representative. At the time of his death he was sales representative in the Kalamazoo area for the Noble & Wood Machine Co. of Hoosick Falls, N. Y. He was a member of TAPPI.

He is survived by his widow, Mildred W. and a sister Mrs. Lloyd Walker.

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TAPPI DIVISIONS AND COMMITTEES

Recent Reports of Activities

Paper-Plastics Conference

The TAPPI Paper-Plastics Conference will be held in Marshall Hall, New York State College of Forestry, Syracuse, N. Y., on Oct. 19–20, 1950. E. C. Jahn, Chairman of the TAPPI Plastics Committee is General Chairman.

Room reservations should be make direct with the Syracuse Hotel or Hotel Onondaga, Syracuse, N. Y.

Following is the final program:

Thursday Morning, October 19, 9:30 a.m.

I. Properties of Plastics in Relation to Papermaking

Discussion Leader—Herman Mark, Polytechnic Institute of Brooklyn

1. "New Polymers and Their Possible Application to Paper," by Herman Mark, Polytechnic Institute of Brooklyn.
2. "Adhesion Fundamentals," by D. B. Hatcher, Plaskon Division, Libbey-Owens-Ford Glass Co.
3. "Permeability of Polymers to Vapors," by A. D. McLaren, Polytechnic Institute of Brooklyn.
4. "Effect of Plasticizers on Physical Properties of High Polymers," by R. S. Boyer, The Dow Chemical Co.

Lunch—Drumlins, 12:30 p.m.

Thursday Afternoon, October, 19, 2:00

II. Beater Addition Fundamentals

Session Chairman—C. E. Libby, New York State College of Forestry

Discussion Leader—Allan Schenck, Riegel Paper Corp.

1. "The Effect of Latex Variables in Beater Addition of Nitrile Latices," by D. M. Yost, Sorg Paper Co., and William H. Aiken, The Goodyear Tire and Rubber Co.
2. "The Effect of Certain Papermaking Variables in Beater Addition of Nitrile Latices," by D. M. Yost, Sorg Paper Co., and William H. Aiken, The Goodyear Tire and Rubber Co.
3. "Phenolic Resins in Paper Modified by Acrylonitrile Rubbers," by R. C. Bascom, B. F. Goodrich Chemical Co.
4. "Headbox Chemistry," by V. J. Moser and I. J. Gruntfest, Rohm and Haas Co.

Friday Morning, October, 20, 9:30 a.m.

III. Cellulose—Accessibility, Reactivity, and Properties Relating to Plastics

Session Chairman—F. A. Simonds, U. S. Forest Products Laboratory

Discussion Leader—John A. Howsman, American Viscose Corp.

1. "The Accessibility of Cellulose," by Harold Tarkow, U. S. Forest Products Laboratory.
2. "Kinetics and Equilibria in Cellulose Reactions," by H. M. Spurlin, Hercules Powder Co.
3. "Accessibility as Determined by Formic Acid Esterification," by R. F. Nickerson, Monsanto Chemical Co.
4. "Pulp Property Requirements for Dissolving Pulps," by Robert H. MacClaren, Eastman Kodak Co.

Lunch—Drumlins, 12:30 p.m.

Friday Afternoon, October, 20, 1950

IV. Plastic Coating Fundamentals

Session Chairman—R. T. Nazzaro, New York State College of Forestry

Discussion Leader—R. H. Kienle, American Cyanamid Co.

1. "Alathon Polythene Coatings—Properties and Techniques," by Alfred Stockfleth, E. I. du Pont de Nemours & Co.
2. "The Effect of Compounding Variables on Styrene-Butadiene Latex Used for Protective Coatings for Paper," by M. E. Wendt and William H. Aiken, The Goodyear Tire and Rubber Co.
3. "The Technology of Styrene Latex Paper Coating," by Eli A. Haddad, Monsanto Chemical Co.
4. "The Versatile Vinyl Enters the Paper Coating Field," by A. L. Hatfield, B. F. Goodrich Co.

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LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel and Events

Officers of Local Sections

The meetings of the local section are held each year from September to June inclusive. At the May or June meetings officers are elected to carry on the administrative activities of each section for the ensuing year. The officers who were elected for the 1950–1951 terms are as follows:

Pacific

Chairman—Robert I. Thieme, Soundview Pulp Co., Everett, Wash.
Vice-Chairman—Eric O. Ericcson, Puget Sound Pulp and Timber Co., Bellingham, Wash.
Secretary-Treasurer—Robert M. True, General Dyestuff Corp., Portland, Ore.
Executive Committeemen—Officers and Walter F. Holzer, Crown Zellerbach Corp., Camas, Wash.; Fred J. Weleber, Publishers, Paper Co., Oregon City, Ore.; Svarre E. Hazelquist, Pulp Division, Weyerhaeuser Timber Co., Longview, Wash.; and Walter A. Salmonson, Draper Bros. Co., Portland, Ore.
Past-Chairmen—C. P. R. Cash, R. S. Wertheimer, C. W. Morden, R. B. Hauser, Lawrence Killam, M. W. Black, W. R. Barber, Carl Fahlstrom, Carl E. Braun, E. P. Wood, C. A. Enghouse, Erik Ekholm, H. W. Bialkowsky, G. H. Gallaway, J. L. McCarthy, H. C. Wall, and W. F. Holzer.
National Executive Committee Representative—Harold W. Bialkowsky, Pulp Division, Weyerhaeuser Timber Co., Longview, Wash.

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Vice-Chairman—A. P. Adrian, Neenah Paper Co., Neenah, Wis.
Secretary-Treasurer—H. W. Rowe, Nekoosa-Edwards Paper Co., Port Edwards, Wis.
Executive Committeemen—Officers and G. R. Sears, Institute of Paper Chemistry, Appleton, Wis.; Thomas C. Catlin, Gilbert Paper Co., Menasha, Wis.; and John C. Wollwage, Kimberly-Clark Corp., Neenah, Wis.
Past-Chairmen—G. K. Hill, Otto Kress, R. J. Le Roux, A. T. Gardner, S. D. Wells, J. R. Fanselow, H. W. Morgan, E. H. Voightman, R. A. Nugent, M. L. Downs, N. L. Malcove, J. H. Graff, T. R. Probst, J. P. Weidner, H. P. Dixson, L. V. Forman, C. J. West, Jr., A. M. Heald, and G. R. Sears.
National Executive Committee Representative—Martin L. Downs, Thilmany Pulp and Paper Co., Kaukauna, Wis.

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First Vice-Chairman—Arthur J. Haug, Scott Paper Co., Chester, Pa.
Second Vice-Chairman—H. C. Brill, E. I. du Pont de Nemours & Co., Inc., Newport, Del.
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Treasurer—F. J. Lovegren, W. C. Hamilton & Sons, Miquon, Pa.
Executive Committeemen—Officers and J. P. Weidner, Container Corp. of America, Manayunk, Pa.; and John Macadam, National Vulcanized Fibre Co., Yorklyn, Del.
Past-Chairmen—W. R. Maull, G. E. Landt, H. P. Connor, H. C. Schwalbe, J. d'A. Clark, C. M. Connor, E. J. Albert, J. D. Davis, W. M. Shoemaker, A. M. L. Bixler, John Macadam, and J. P. Weidner.
National Executive Committee Representative—Ralph W. Kumler, American Cyanamid Co., New York, N. Y.

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Vice-Chairman—H. A. Smith, Mead Corp., Chillicothe, Ohio.
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Corresponding Secretary—Paul Clark, Crystal Tissue Co., Middletown, Ohio.
Treasurer—Robert D. McCarron, Stein, Hall & Co., Cincinnati, Ohio.
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National Executive Committee Representative—Kenneth P. Geohegan, Aetna Paper Co. Div., Howard Paper Mills, Inc., Dayton, Ohio.

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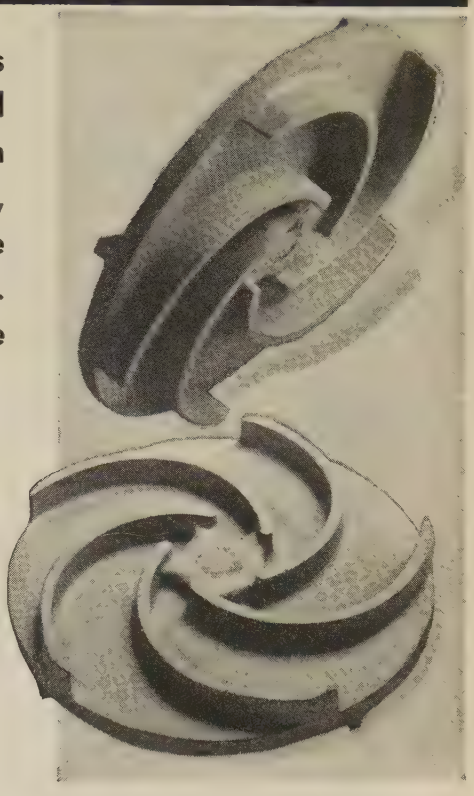

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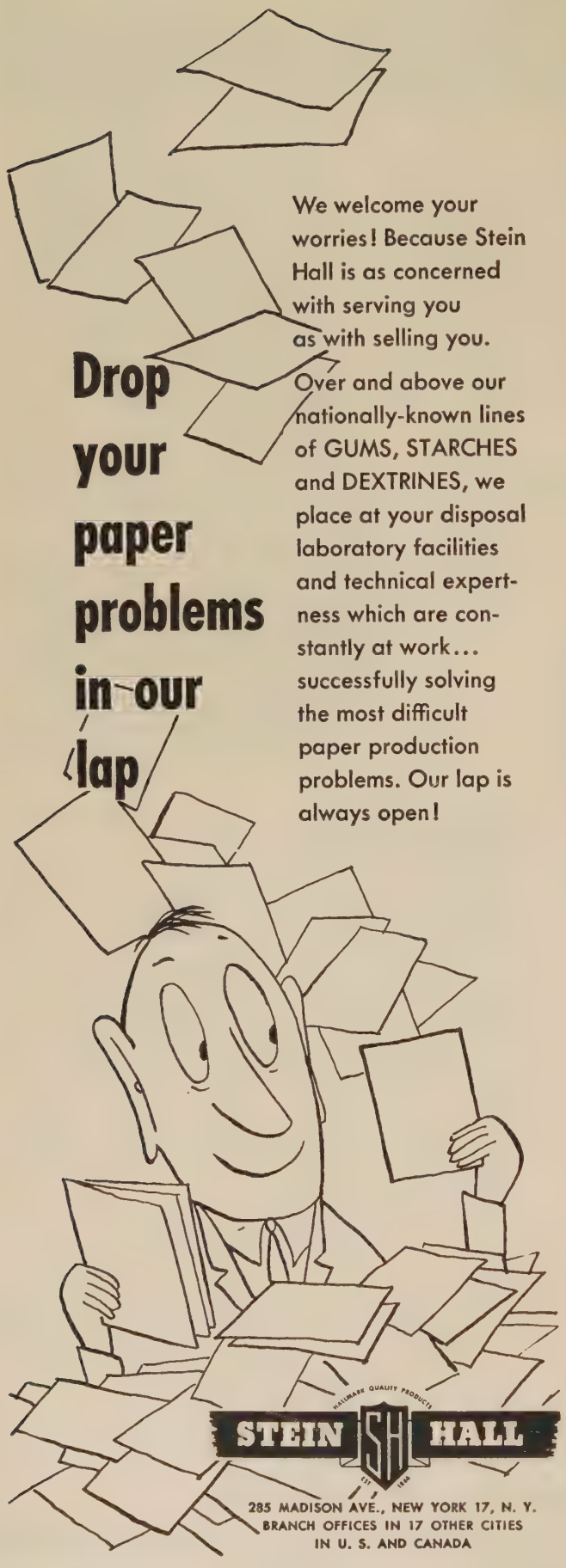
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Vice-Chairman—Wm. Birdsie, Flintkote Co., Los Angeles, Calif.

Secretary-Treasurer—Joseph Cooey, Angelus Paperbox Co., Los Angeles, Calif.

Executive Committee—Walter Quinn, Container Corp. of America, Los Angeles, Calif.; Arthur Kane, Westinghouse Electric Corp., Los Angeles, Calif.; and Robert W. Stevens, Consultant, Monterey Park, Calif.

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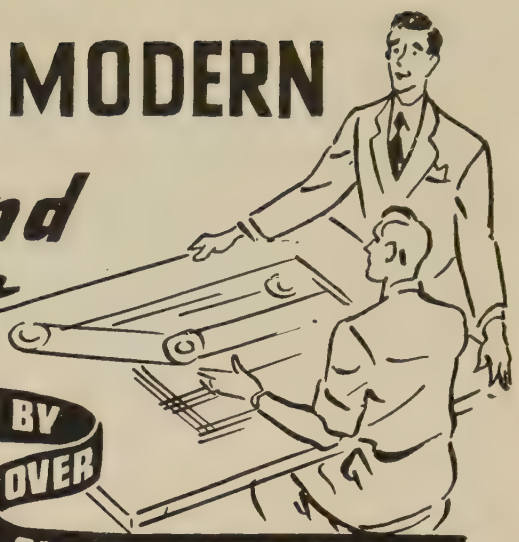

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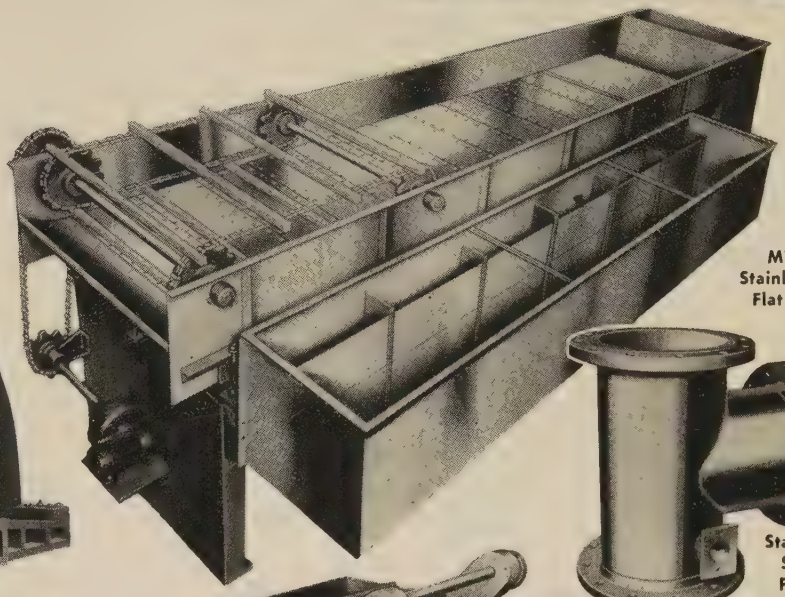

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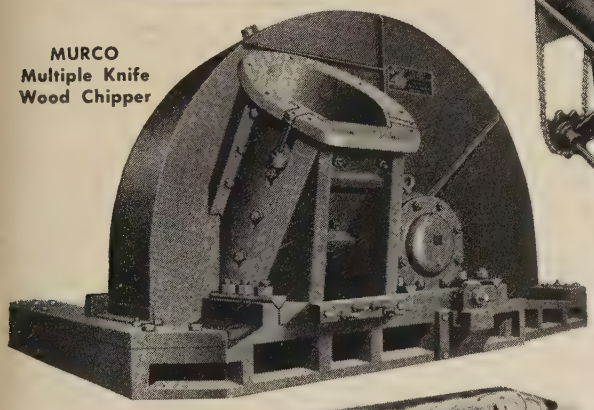

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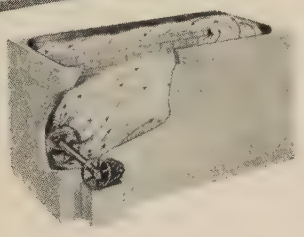

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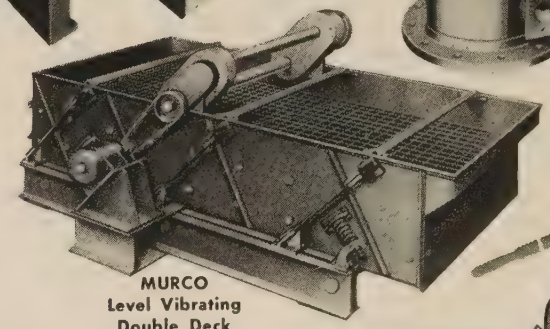

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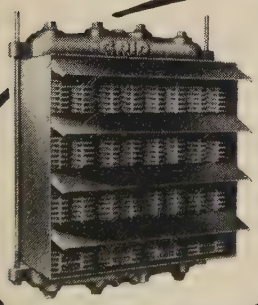

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Progress in Alkaline Pulping in 1949

SIDNEY D. WELLS

The review of the progress in alkaline pulping in 1949 that follows is a continuation of the corresponding reports for 1946, 1947, and 1948 (*1*). There seems to be no tendency as yet for the rate of expansion to slacken, even though such expansion in new locations entails expenditures of 20 to 40 millions of dollars for the consumation of a well-integrated plant. Joint ownership by pulp manufacturer, board manufacturer, and converter or by pulp and paper manufacturer and publisher is becoming increasingly common.

Substantially all the increased production of long-fibered pulp demanded by the expanding paper industry is made up largely by new production by sulphate pulping. Uses that do not require long fibers are supplied by the semichemical neutral sulphite process, the inauguration of which occurred at the Forest Products Laboratory in 1923. Where sulphate pulping and semichemical neutral sulphite pulping occur adjacent to each other, cross recovery makes possible the elimination of stream pollution and the use of all species of wood within reasonable shipping distance; the increased average yield of pulp from both processes amounts to 60%, instead of the 45% previously realized by established practice. With the routing of all coniferous woods to the kraft operation and all hardwoods to the semichemical neutral sulphite operation, the pulps can be utilized in the widest range of products with yields, strength, and brightness at whatever level is demanded consistent with existing knowledge of fundamentals. Multistage chlorination and bleaching, established in the early 1930's has extended the use of kraft pulp to all grades of paper and board and in the late 1940's the same procedure was extended to semichemical pulps. In 1924 this possibility was demonstrated in pilot-plant operation and the recent plant-scale experience has confirmed the soundness of the early work.

GENERAL

Jayme (*2*) reviewed the literature published in Germany during the war years. The annual directory of the mills operating in the 14 southern states and Puerto Rico has made its appearance (*3*). The reported production (*4*) of unbleached kraft is 2,200,000 tons. In 1949 1,900,000 tons were reported or a decline of 12% (*5–7*). The production for 1950 will probably equal that of 1948. Standard terms for the industry have been recommended and presented by the Canadian Pulp and Paper Association (*8*). A limited edition of 115,000 copies of its report commemorated the 50th anniversary of the founding of the International Paper Company (*9*). Of particular interest are the kraft paper and board operations, with a daily capacity of over 4000 tons. Gillespie (*10*) discussed the trends in the development of the sulphate industry in the South.

EXPANSION IN THE UNITED STATES

Modernization of existing plants is contributing greatly to the increasing capacity of the industry in the United States (*11*). The Sonoco Products Co. is an interesting example (*12–15*). The original company, the Carolina Fibre Co. at Hartsville, S. C., commenced operation at the start of the 20th century and established the manufacture of sulphite pulp and groundwood pulp from southern pines and gums. By the middle 1920's the Sonoco Products Co. was organized, based upon a cylinder machine operation; about 20 years later the two operations were consolidated into one, making possible a wide assortment of fourdrinier and board machine products. In the late 20's the semichemical neutral sulphite process was found to be better suited than the sulphite process for the production of the pulp which was required and now, with the collaboration of the Ohio Boxboard and the Hankins Container Companies, a greatly expanded operation and modernization is under way. A 300-ton sulphate pulp mill for the production of dissolving pulp from southern hardwoods is under construction at Natchez, Miss. (*16*). The Oxford Paper Co. of Rumford Falls, Me. is modernizing their mill which manufactures 600 tons of book paper per day. A C-E recovery furnace and boiler and 6 welded digesters, one of which is of stainless clad, are among the improvements (*17*). In Wisconsin, both the Thilmany Pulp and Paper Co. and the Nekoosa-Edwards Paper Co. have extensive modernization programs nearing completion (*18,19*). At International Falls, Minn. similar expansion improvement and modernization have taken place (*20–23*).

In Georgia the Brunswick Pulp and Paper Co. has greatly enlarged its production capacity and improved its operation (*24*). A Cottrell precipitator contributes to the latter. At Macon, the Macon Kraft Co. (*25–30*) has gone into operation, making 600 tons of kraft per day for Inland Container Corp. and Mead Corp. All this production is made on one huge fourdrinier paper machine. At Coosa Pines in Alabama, the Coosa River Newsprint Co. is now in full production. The product has been used by northern as well as southern newspapers. The skepticism with which the paper industry and newspaper publishers have viewed the use of southern woods for paper manufacture during the past 50 years has now been shown to be largely unfounded and, with the growth in the number of personnel skilled in the art, a substantial portion of our paper, pulp, and board requirements now flow from this region (*31, 32*).

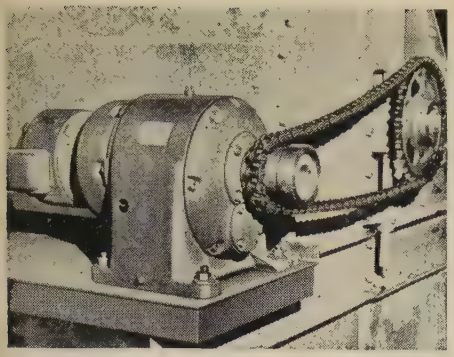
On the Pacific Coast the Tacoma, Wash. mill of the St. Regis Paper Co. has been modernized, enlarged, and completely integrated by the addition of a paper mill to the bleached pulp operation (*33, 34*).

The Crown Zellerbach mill at Camas has built a new wood room so as to bark and chip West Coast logs (*35*). The Crown Zellerbach mill at Port Townsend has been modernized in the pulp washing, screening, and recovery operations (*36*). The papermaking facilities have been in a continual state of modernization. The Longview Fibre Co. has installed a C-E recovery furnace and boiler (*38*). Integration of pulp manufacture with logging and lumber manufacture is well accomplished by Weyerhaeuser at Longview, Wash. and Springfield, Ore.; the kraft pulp mills and a board mill on the latter site are described (*39–46*). Three hundred and fifty tons of bleached and unbleached pulp per day from Douglas-fir logging and mill waste have thereby come into being. Another mill closely associated with the Weyerhaeuser operations is that of Potlach Forests, Inc., under construction at Lewiston, Idaho (*47*).

EXPANSION IN CANADA

The expansion characteristic of the industry in the United States is also true of that in Canada. Many new mills have been built and gone into production and many long established mills have been modernized and expanded (*48*). A 150-ton kraft mill has been built at Newcastle, N. B. to supply pulp for bleaching and conversion into paper at the Fraser Companies, Ltd. paper mills at Edmundston, N. B. and Madawaska, Me. (*49, 50*). The Bathurst Power and Paper Co. (*50, 51*) has expanded and modernized the mill at Bathurst, N. B. The same is true for the Howard Smith paper mills in the Province of Quebec and the mills of the Brown Corp. at La Tuque, Que., which make 450 to 500 tons of bleached kraft, much of which is well known throughout the world under the name of Cellate (*52, 53*). The conversion of the obsolete and long idle newsprint mill at Espanola, Ont. by the Kalamazoo Vegetable Parchment Co. to an up-to-date kraft mill with newsprint paper as a side line is a shining example of how a ghost town can be revived,

SIDNEY D. WELLS, Member, TAPPI; Research Associate, The Institute of Paper Chemistry, Appleton, Wis.



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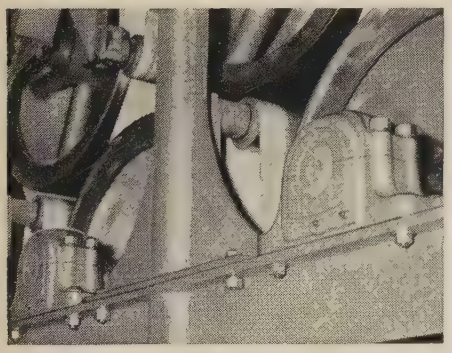

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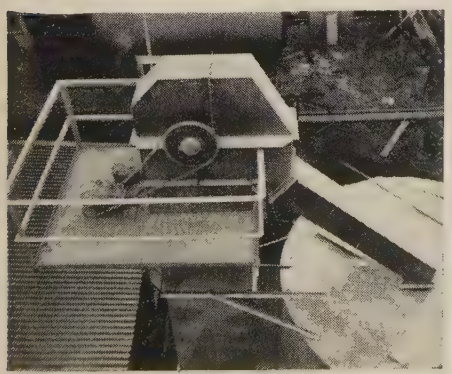

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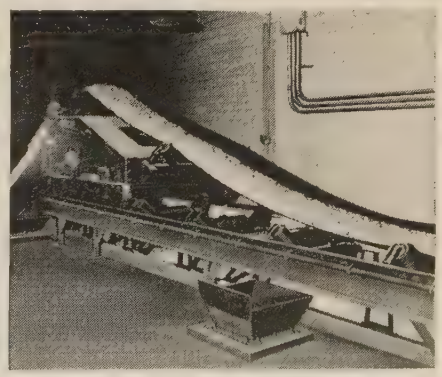

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exhausted timber limits made productive, and a decadent property revived and made to create wealth adequate for the support of the whole operation and a source of pulp for a pulp-hungry industry in the United States. With the power not needed for the pulp and paper operation, the chemicals for pulping and bleaching are produced electrolytically (*54*). A similar transformation has been effected by the Abitibi Power and Paper Co. in the conversion of the sulphite and newsprint mill at Sturgeon Falls into a 100-ton semichemical neutral sulphite corrugating paper mill making 32,500 tons of paper products per year from species of wood left behind in the defunct newsprint operation (*55–58*). This, together with the spread of the use of the process in the United States, Europe, Japan, and Australia, is a notable example of the returns from applied research at the Forest Products Laboratory in the 1920's for the betterment of humanity (*59–63*).

Probably one of the outstanding developments of the past two decades is the adoption of the kraft process for the pulping of northern conifers for the production of bleached high-strength pulps for which the sulphite process was heretofore used. The mills at La Tuque, Marathon, Espanola, Red Rock, Tacoma, Lewiston, Newcastle, Bathurst, and Nekoosa are illustrations. An outstanding example is the LongLac mill at Terrace Bay, Ont. (*64–68*). Ninety per cent of the steam required for operation is obtained from the burning of waste, particularly the organic material in the spent cooking liquor, which otherwise would pollute the adjoining river and lake front (*69*). Between 1910 and 1918, Moore and co-workers (*70*) established that, by concentration and burning of the spent liquors under proper conditions, sufficient heat was evolved to not only supply the requirements of the recovery operation but also to furnish all the steam for cooking and washing and a substantial portion of that required for drying the pulp. Rosencrants (*71*) further simplified the operation and established the merits of water-tube boilers with gas flow parallel to the tubes in vertical settings. The Marathon operation in Ontario is further discussed (*72*).

Much activity has occurred in British Columbia (*73*). On Vancouver Island the Nanaimo Sulphate Pulp Co., with a planned production of 200 tons of bleached kraft pulp per day, is nearing completion (*74–78*). Plans for the improvement and modernization of the Sorg Pulp Co. at Port Mellon have been announced (*79*).

OTHER COUNTRIES

In Scotland the 100-year old operation of the Clyde Paper Co. Ltd. is described, with particular mention of the use of esparto as the main fiber source for the two paper machines and coating equipment (*80*). The present state of the kraft paper industry in Finland is discussed (*81*). In Algiers at Baba Ali, the mill of Compagnie Nord-Africaine de Cellulose has been completed and the company has commenced manufacturing operations, using a continuous process throughout. The Cellulose Development Corporation Ltd. supervised the layout, making use of the Celdecor-Pomilio-Kamyr pulping and bleaching systems. Esparto and straw are the fibrous raw materials (*82, 83*). In India the Bengal Paper Mills Co., pioneers in the use of bamboo and the crusher for pulping bamboo, are erecting a new plant, including a recovery and waste heat boiler, for pulping sabai grass (*84*). A newsprint mill is under construction at Chandru (*85*). At Dalmianagar in Bikar, a fine paper mill is being built; the operation is based upon the use of bagasse pulped by the Celdecor-Pomilio process (*86*).

CONSUMPTION OF CHEMICALS

Summaries and reports on the consumption of nonfibrous raw materials, particularly chemicals, appear each year since the paper industry is an important consumer of some of them (*87–90*); from many angles it is also a chemical industry (*91*). It is second in the use of sulphur (*92*). Alkali and chlorine are the most important chemicals for pulping, pulp purifica-

tion, and bleaching (*93–96A*). With the combination of sulphur with the hydrogen evolved in the production of chlorine and caustic soda, sodium sulphide may be produced so as to thoroughly integrate an electrolytic plant with the operation of a bleached kraft mill (*97, 98*).

WOODS USED

Pulp and paper mills are becoming more self sufficient in regard to their wood supply and the building up of forestry projects capable of a sustained yield production of pulpwood. Production by local farmers is also encouraged (*99–105*). With favorable conditions and rapid growth, the South is advantageously situated (*106–111*). Considerable activity in organization is underway on the West Coast (*112*) and also in Canada (*102, 113*). Aspen (*114*) poplar, and willow (*115*) have come into their own as pulpwoods, as have oak and chestnut oak (*116, 117*). Various committees of the United Nations are making surveys and appraisals of the pulp and paper situation from a world standpoint (*118, 119*). Much has been presented as a result of the fourth meeting of the Technical Committee on Wood Chemistry of the Food and Agriculture Organization (*120–123*). The Yearbook of Forest Products Statistics is also an important contribution (*124*). Harrison (*125*) discussed the world supply of 40,000,000 cubic feet of softwood annually which is accessible and 20,000,000 cubic feet which is inaccessible. Atchison (*126*) discussed the present and potential use of hardwoods in Western Europe.

PULPING PROCEDURES FOR VARIOUS SPECIES

Proper integration of the wood-using industries of northern Michigan from the logging and saw mill waste would make possible a corrugating paper, newsprint, and book paper operation of sufficient magnitude to be competitive with operations in established locations and a factor in the industry (*127*). Hardwoods in southern New England offer a similar opportunity (*128*). Experiments on scrub oak in Florida indicate its possible utilization for corrugating materials (*129*). Researches on poplar in Germany (*130*) and on beech, using prehydrolysis followed by kraft cooking (*131*), are reported. In Japan birch occurs and is being studied as a source of pulp (*132*). The interest in the semichemical process continues, with articles appearing from many parts of the world (*133–140*). Aspen is most commonly used in the application of the process for making pulps for printing and better grades of paper (*141*). Use of digestion without chemicals is also practical for coarse products (*143*). Considerable work is in progress in Australia on the use of eucalyptus (*144–146*). Revival of interest in the hydrosulphide process, developed by Viggo Drewsen in the early years of the century, has occurred in Japan (*147, 148*). Jayme and Schenck (*149*) report on the effect of various alkaline purification procedures on the behavior of the resulting pulps on acetylation. Enkvist, Moilanen, and Alfredsson (*150*) report on the yield and reaction velocity in the formation of thiolignin in the presence of hydrogen sulphide. Murto (*151*) discussed the use of sulphur with white liquor for cooking birch and pine. Equchi (*152*) discussed the effect of cooking to various lignin contents and with various amounts of active alkali and sulphidity on the pulp produced. Martin (*153*) reported studies on the kraft pulping of Douglas-fir, and Hammond and Billington (*154*) have studied the effect of various rates of growth and ages of this wood. Extensive studies have been made on the pulping characteristics of Douglas-fir, western hemlock, western red cedar, eastern black spruce, and loblolly pine. Holzer and Booth (*155*) reported on their pulping characteristics; Lewis, Wise, and Browning (*156*) on the chemical characteristics of the unbleached pulps; Holzer and Lewis (*157*) on the relative physical properties of the pulps from the spring- and summerwoods of Douglas-fir; Browning and Baker (*158*) on the relation between strength development and specific surface

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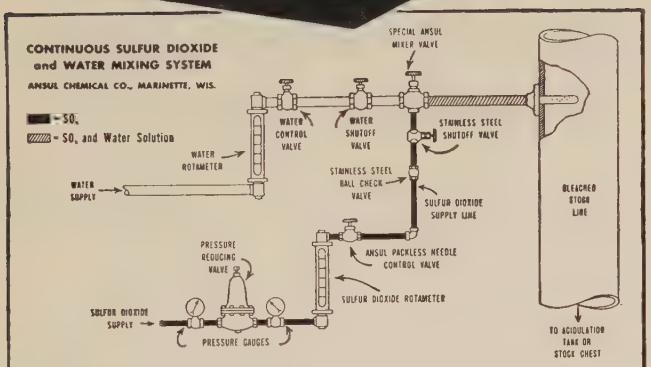

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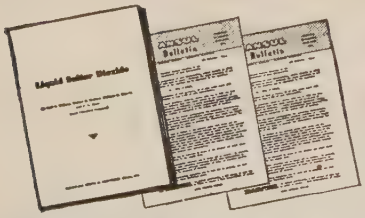

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of the unbleached pulps of the various species mentioned; Clark (*159*) on the x-ray diffraction orientation; Graff and Isenberg (*160*) on fiber lengths; and Heuser, Shockley, and Kjellgren (*161*) on the chain-length distribution and characteristics.

AGRICULTURAL RESIDUES AND GRASSES

Many fibers in addition to wood are used for paper—in fact, wood is relatively a newcomer in the field. Cotton and linen have been used for many centuries; use of esparto is as old as that of wood, and straw dates back to the first century of the Christian era. The recent war cut off the supply of esparto for a time but its use is now substantially as great as before the war (*162–164*). Establishment of new plants elsewhere than in Great Britain is a possibility (*165, 166*), as is shown by the new mill in Algiers (*82, 83*). This mill also pulps straw and similar plant materials are in use all over the world (*167*).

Atchison (*168*) reported on the use of straw and other agricultural residues in the United States. Grothaus (*169*) reported that large quantities of straw are available in Kansas and Meisen (*170*) stated that power will also be available in North Dakota in the next few years with plenty of straw—both cereal and flax. Work on the chemical constitution of straw has been reported by various authorities. Delga (*171*) gave the general composition and the variation from year to year and place to place has been also studied (*172*); Adams and Castagne (*173*) determined the distribution of methoxyl and uronic acid groups and Ko and Hilpert (*174*) studied the xylan content. Gladkikh (*175*) discussed the wide use of straw by the paper industry. Bertrand and Silberstein (*176*) described new researches concerning the manufacture of straw paper by maceration. Dreyfus (*177*) disclosed the treatment of straw with dilute (0.5 to 2.5%) caustic soda at 100 to 125°C., followed by mechanical disintegration and treatment with 10 to 20% hydrogen peroxide solution and 7 to 20% caustic soda at 50 to 60°C.

A complete report of the fall meeting of the TAPPI Committee on the Use of Fibrous Agricultural Residues, held at the Northern Regional Laboratory Nov. 15–16, 1948 has appeared (*178*). Aronovsky (*179*) brought out the relation of straw to other pulps in paper manufacture. Lathrop (*180*) discussed the merits of wheat straw for improved paper and board products. Aronovsky and Lathrop (*181*) described the use of the Hydrapulper at temperatures near 100°C., using kraft cooking liquor for pulping agricultural residues (*181*). Lathrop and Naffziger (*182, 183*) discussed the evaluation of agricultural residues for structural board products. Krancher (*184*) commented on the revival of interest in straw paper manufacture, with the use of caustic soda and sodium sulphite in place of at least a part of the lime for pulping, and the interest in the Morley continuous pulper and the Hydrapulper. Krancher and Price (*185*) reported on mill runs using the Hydrapulper. Root (*186*) gave additional data on its use. Lamb (*187*) prepared a résumé of the factors to be considered and the present practice in pulping straw and stock preparation and Bush (*188*) discussed some of the practical aspects. Herman (*189*) discussed the use of the roll-type sugar cane mill.

Belford (*190*) discussed a farmers' market for straw, Adair (*191*) straw procurement by the Alton Boxboard Co., Lyon (*192*) the benefits of educational effort on the procurement and quality of straw, Stahl (*193*) the merits of preservatives for the storage of straw, and Wilson (*194*) the effect of single vs. multifill loading of rotaries Textor and Eberhardt (*195*) discussed the use of revolving disk mills for pulping and refining straw, Ritman (*196*) compared four methods for manufacturing straw pulp, and Merrill (*197*) reported on the use of sodium silicate. Timlowski and Kinsley (*198, 199*) describe the trial in a Holyoke paper mill of straw pulped with a mixture of conventional cooking alkali and 6 to 10%

of an organic inhibitor of undisclosed composition on a commercial paper machine in the manufacture of newsprint.

Relier and Marpillero (*200, 201*) discussed pulp from straws, other annual plants, and grasses; Atchison (*202, 203*) surveyed the lime, caustic soda, kraft, sodium monosulphite semichemical, Marsoni, Huguenot, and Celdecor-Pomilio processes as used commercially in Europe and elsewhere in making board and high-grade papers, and the present and potential utilization of straw in Italy, Western Germany, The Netherlands, Belgium, France, Austria, and the United Kingdom.

Eguchi and Suzuki (*204*) discussed the use of rice straw, and Kung (*205*) cellulose from annual crops, especially cornstalks. Sweeney and Arnold (*206*) reported the effect of moisture in the manufacture and use of cornstalk insulating board. Lewis (*207*) showed how the fractionation of pulps from annual plants, as disclosed by Wells (*208*), makes it possible to obtain a bleached, long, fine-fibered pulp capable of use in high-quality papers without imparting the usual lack of opacity and the brittleness heretofore characteristic of attempts to utilize pulps from agricultural residues, annual plants, and grasses, and to utilize the non- and short-fibered fraction with cheap waste paper stock, insulating board stock, and similar material as a stiffening agent to impart crushing strength, rigidity, and high bending resistance to corrugating paper, boxboard, structural wallboards, and hard board. By producing co-products, the cost of each is materially lowered and the economics is vastly improved.

West (*209*) published a supplement to his annotated bibliography on the utilization of bagasse for paper, paperboard, plastics, and chemicals. Litkenhous (*210*) reported on War Production Project 134 for the utilization of bagasse for paper, board, fillers, etc., bringing out the facts that the possible value may often exceed the value of the sugar. Wiggins (*211*) stressed the importance of research for bringing about the more complete and profitable utilization of products from the sugar industry. The *Chemurgic Digest* (*212*) gives a general review of the use of bagasse and the *Indian Pulp and Paper* (*213*) discussed the wealth possible from the utilization of bagasse. Fuller (*214*) disclosed a tower for drying bagasse.

Okada and Shogenji (*215*) described the pulping of kozo (the best fibers of paper mulberry) at 100° with 1 to 15% sodium hydroxide on the weight of the fiber for 0.5 to 50 hours. Sproull (*216*) and Sproull and Banks (*216A*) showed that *Lespedeza servicea*, grown in the South for soil improvement, can be used as a source of papermaking fiber. Dixon (*217*) reviewed the use of the fiber from seed flax straw for cigarette paper; this application was inaugurated in 1937 and now amounts to 60,000 tons of decorticated fiber annually. The shive is used also for fuel, wallboard stock, feed, and bedding. Williams (*218*) discussed the products from flax straw. Tobler (*219*) discussed the lignification of bast fibers. Cook (*220*), Patterson (*221*), and Reeves (*222*) disclose portable machines for the decortication of flax straw, hemp, sansevieria, henequen, and the like. Angibaud (*223*) discussed the utilization of various colonial plants, Tomeo, Mene, and Herrero (*224*) discussed matweed, flax straw, and flax tow, and Allquist (*225*) disclosed the use of ordinary industrial water acidified with inorganic mineral acid to pH of 2 to 5, followed with demineralized water for rinsing, for washing pulp. White and Hawker (*226*) disclosed a machine for the separation of the bast fibers from remie and similar materials. Berkman (*227*) discussed the use of milkweed grown on submarginal lands. Patettor, Kumayay, Vay, Rubi, and Capo (*228*) reported on the use of Paraguayan cane as a raw material for paper pulp. Considerable interest has developed and the possibilities studied for utilizing the enormous and luxuriant growths of cane in the Danube delta. Radulesscu (*229*) and Barbasche (*230*) discuss the projects under consideration. Tomeo and Mene (*231*) do likewise.

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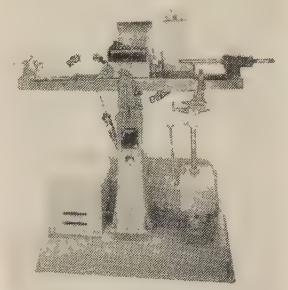

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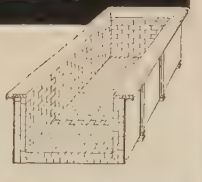

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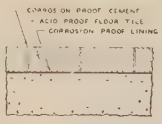

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Chittenden and Coomber (*232, 233*) report on banana fiber from Jamaica and castor stems from Ceylon. Cruse (*234*) discussed pulp from yucca and other desert flora.

COOKING PROCEDURES AND DIGESTOR FACILITIES

Prehydrolysis of wood prior to alkaline digestion has recently received much attention, especially in Germany during the war years, as a method for the preparation of furfural and sugars. Application to hardwoods showed considerable merit where conditions warranted. Applied to the softwoods the loss of yield and frequency of strength of the final pulp has discouraged extended use. Ullmann (*235*) disclosed the practice in a Canadian patent. Hägglund and Webjörn (*236*) discuss various mild delignification treatments that seem to indicate that the presence of polyoses of low molecular weight are important to the beating properties and that lignin residues may cloak and retard their influence. Hägglund (*237*) also discussed the speed of lignin dissolution and showed why thiolignin dissolves more rapidly. Ueno (*238*) reported that hydrosulphide lignin is lower in methoxyl than sulphite lignin. Enkvist and Hägglund (*239*) discuss sulphidation, methylation, and sulphonation of lignin. Eguchi and Ito (*240*) discuss the relationship of the degree of cooking to pulp strength; the tensile and bursting strengths are highest in a pulp with a Roe number of 10 to 12; tearing strength increases with prolonged cooking. Richter (*241–243*) disclosed a continuous digester with indirect heating, discussed the possibilities of the idea, and reported on a pilot-plant operation capable of producing 8 tons per 24 hours with a steam consumption of less than 2000 pounds per ton. Sloman (*244*) disclosed a two-stage countercurrent process for cooking with the pH of the black liquor after the first stage less than 10.2. McAlear (*245*) disclosed a digester gasoff, strainer cleanout, and control mechanism with pressure and temperature responsive elements. Wells (*246*) patented a method of cooking using impregnation and continuous injection with conductometric control so as never to exceed or be below certain limitations which assure higher yields, better bleachability, reduced chemical consumption, higher degree of polymerization, and greater strength. Carlander (*247*) described the use of caustic soda and sulphur. The U. S. Forest Products Laboratory (*248*) reported on the Conference on Semichemical Pulping; McGovern, Mackin, and Chidester (*249*) and Chidester (*250*) discuss semichemical pulping of hardwoods with high yields and good strength properties of the resulting pulps.

Asplund (*251*) disclosed pretreatment with alkali, pressing, and defibration. Kehoe (*252*) and Beveridge and Kehoe (*253*) disclosed alkaline pretreatment with defibration. Goodwin (*254*) discussed recent installations of the Chemipulper. Libby and O'Neil (*255*) describe a chemigroundwood process similar to the Fish process or the modified boiled groundwood processes studied in the 1920's. Hurter (*256*) described an improved system for chip storage, blending, and conveying and Swift (*257*) disclosed the hydraulic barking of wood. Hummel (*258*) described magnetic separation systems to eliminate tramp iron in chips, stock lines, etc., ahead of disk refiners, jordans, and other places that cannot tolerate it.

FOAM

Sergeant discussed foam troubles and foaming (*259*) and Young (*259A*) disclosed improvements in foam reduction during washing.

TALL OIL AND TURPENTINE RECOVERY

Hasselstrom (*260*) discussed the biogenesis of the resin acids from pine tree resenes. Pollak (*261*) discussed the composition, grades available, and applications of tall oil. Rumpel (*262*) considered tall oil as a raw material in the chemical industry. The Technical Association of the Pulp

and Paper Industry (*263*) proposed standards for tall oil skimmings. Demarcq (*264*) lists 86 references in a review of tall oil and its uses. Snell and Reich (*265*) discuss the physical-chemical properties of tall oil soap solutions and De Bartholomaeis (*266*) gives an evaluation of this material. The Tall Oil Association (*267*) reported that probably 30,000,000 pounds of tall oil are used in soap products annually, in contrast to the 16,000,000 pounds reported in the June 30, 1949 Bureau of Census figures. Complete recovery of the tall oil soap present in black liquor and substances such as pinosylvin may greatly reduce the deleterious effects on shellfish (such as oysters) of the effluents from kraft pulp mills (*268*). Jakobson (*269*) reviews the literature on tall oil between 1935 and 1945. Juvonen (*270*) discussed the present status of the tall oil industry in Finland; this started at the Kotka mill in 1920. Stenerson (*271*) discussed the position of tall oil in the chemical markets. The National Southern Products Corp. (*272*) presented an annotated bibliography of possible applications for tall oil. The Tall Oil Association (*273*) has issued three bulletins covering testing methods, use in drying oils, and use in soap products. The National Southern Products Corp. (*274*) has issued seven bulletins covering general information, pertinent data, various uses, etc. Young, Eick, and Warmack (*275*) point out that tall oil is now a product which has been stabilized as to constituents and is undergoing improvement by removal of color bodies, sulphur compounds, etc. Heinanen (*276*) discussed its autoxidation. Thurman (*277*) disclosed a method for concentration of black liquor and the removal of the soap by centrifugal action; this is similar to Drewsen's patent 1,778,523 issued in 1930.

Bolley (*278*) discussed synthetic drying oils, Saracini (*279*) the use of tall oil in varnish manufacture, and Emerson (*280*) the technology of the tall oil paint and varnish industry. The Northwestern Paint and Varnish Production Club (*281*) presented tall oil varnish-exposure test results and Scheibele (*282*) discussed refining and paint applications. Bent and Peterson (*283*) disclosed methods for producing tall oil resins. White (*284*) described tall oil esters for core bonding and Greenlee (*285*) the esterification of high molecular weight polymers of polyether polyhydric alcohols with tall oil acids.

Khudovekov (*286*) suggested three methods for processing tall oil, Passino (*287*) explained the Solexol process using propane extraction, and Houpt (*288*) disclosed the separation of constituents with secondary amyl alcohol, esterification, and crystallization. Jolly (*289*) proposed the production of nonemulsifiable high-pressure lubricants and greases by the reaction of tall oil nitriles with sulphur at above 190°C., and the treatment with sodium hydroxide to remove sulphur. Koonce and Perry (*290*) proposed the esterification and elution of tall oil for the recovery of valuable constituents. Segessemann and Molnar (*291*) disclosed the manufacture of hard soaps by the degradation of the fatty acids of tall oil to palmitic and myristic acids by treatment with sodium or potassium hydroxide at 230 to 290°C. and 5 to 20 atm. for 6 hours. Patrick and Emerson (*292*) discussed the liquid phase air oxidation of the methyl esters of the unsaturated fatty acids with the formation of mono- and dicarboxylic acids and byproducts of high molecular weight. Enkvist and co-workers (*293–295*) reported on the decarboxylations of tall oil components and pine tar, using various catalysts.

Myers and Muckerheide (*296*) disclosed a cutting oil containing the esterification product of tall oil with propylene glycol. Gordon and Ragot (*297*) discussed the hydrogenation of tall oil under conditions which do not affect the rosin acids and which make a light-colored stock satisfactory for soap. Othmer and Papps (*298*) suggested the recovery of stearic acid and abietic acids from hydrogenated tall oil using nitropropane and petroleum ether or isopropanol as the solvent for eluting the constituents from a column of activated carbon. By heating abietic acid crystals to 280 to 290° in an atmosphere of carbon dioxide, bubbling steam

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through for 80 minutes, holding at 280 to 290° for 3 hours in an atmosphere of carbon dioxide, cooking in an inert atmosphere to 150 to 170°C., and setting in molds, Taylor, Metcalf, and Branscomb (*299*) claimed a product adapted for pale varnishes, white paper size, adhesive tape, etc. Van Valkenburgh (*300*) patented a tall oil acid composition for use in vulcanizing rubber and Barker (*301*) proposed a detergent composition. Draman (*302*) and Kalman (*303*) disclosed methods for producing carbon black for synthetic rubber fabrication. McMurray (*304*), Herkenhoff (*305, 306*), Gieseke (*307*), Weinig (*308*), and De Vaney (*309*) all disclosed means for using tall oil or kraft soaps for agents in the flotation of various ores. Buckley and McCoy (*310*), Dawson (*311*), and Dyke and Self (*312, 313*) disclosed the use of tall oil in asphalt emulsions and drilling muds and fluids. Bogrow and Johnson (*314*) used the resin acid constituents to reduce the viscosity of molten sulphur.

Noll (*315*) gave chemical and physical data on a number of samples of German sulphate turpentine. McGregor (*316*) has proposed a method of purifying sulphate turpentine in which it is shaken with 5 parts of green liquor, decanted, and treated with a bleaching agent.

TESTING METHODS

Levy (*317*) has developed a sampling tool for sampling flax straw in bales. Collins (*318*) compared methods for the determination of sulphide in oxidized kraft black liquors. The Central Laboratory of the Cellulose Industry (*319*) has published a method for the analysis of white liquor. Adams and Castagne (*320*) have proposed methods for the determination of cellulose, lignin, methoxyl, and uronic acid groups in cereal straws. Martin (*321*) developed a quantitive method for the determination of the hydrolysis of sodium sulphide throughout the course of kraft cooking. Brown (*322*) proposed the determination of exposed specific surface of pulp by air permeability measurements. Browning (*323*) compared the TAPPI Standard T 635 m and the formalin modification, the Torrey-Nelson, and the conductometric methods (with correction factors) for black liquor analysis. The TAPPI Tentative Standard (*324*) and the American Society for Testing Materials (*325*) method for the analysis of tall oil skimmings have been issued. Jensen (*326, 327*) described a chromatographic method for the determination of the neutral matter and another for the determination of the free fatty acids in tall oil. Linder and Persson (*328*) have published a method for the determination of the fatty and resin acids by esterification.

RECOVERY OPERATION AND EQUIPMENT

Badger and Lindsay (*329*), in their annual report on evaporation and evaporators, report no startling developments and almost all the references are for operations other than for pulp mill liquors. The Rosenblad system for alternating the steam and liquor sides of the heating surfaces to minimize the effect of scale was not mentioned. Screen mats show some promise of solving entrainment problems. Whether to operate long tube evaporators with or without circulation is still determined by trial rather than by mathematics. Coates (*330*) offered a simplified method of estimating evaporator capacity and steam consumption. Wall (*331*) discussed instrumentation of black liquor evaporators, as well as that for bleach plants. Kobe and McCormack (*332*) reported viscosity values for spent pulping liquors. Bobe and Seip (*333*) discussed the power problems in a modern kraft mill. Edling (*334*) considered the thermo-technical problems in recovery furnaces and boilers. Macklem (*335*) indicated how all the steam power needed may be obtained. Wilcoxson (*336*) disclosed an improved design for recovery furnaces and Broman (*337*) a method for cleaning dry heating surfaces with falling shot. Denisov (*338*) discussed

furnace control and operation from a Russian viewpoint. Gagliardi, Mellor, and Hamm (*339*) disclosed a method for compensating for the fuel value for the soap removed for tall oil production by adding, prior to the furnace, low ash sulphur-containing pulverized coke. Collins (*340*) reviewed the literature on sulphur losses and the oxidation of the unoxidized compounds to reduce losses and odor. Ely and Rogers (*341*) disclosed a spray tower construction for recovery of heat and chemicals from furnace gases. Collins and West (*342*) summarized the experience of the Thilmany Pulp and Paper Co. with the Venturi-Scrubber for recovery furnace gases, multiclone collectors for lime kiln gases, waste product sulphuric acid for make up, and oxidation to reduce hydrogen sulphide odor and sulphur loss. Patterson and Loyd (*343*) related the troubles encountered at Ocean Falls, B. C., from crowding a Tomlinson-Cottrell layout and from sodium chloride in the water-transported and stored pulpwood. Schmidt (*344*) reviewed the fundamentals of Cottrell electrical precipitation and centrifugal collectors. Temperature, humidity, and chemical composition affect resistivity and corrective devices to give good distribution are important. Hand (*345*) reported upon the performance of the Cottrell precipitator at Port Alberni, B. C., naming insulation, electrode rod breakage, corrosion, bridging of deposits, radio interference, and breakdown of high voltage cables as sources of trouble. Different damper construction, high voltage cable protection with high-frequency suppressors at treater ends, and either steel plate walls or grounded heavy wire netting embedded in the concrete are suggested as a shield against radio interference. Coating with Heresite gave gratifying results in protecting against corrosion. Better insulation is needed in cold climates. Roberts and Beaver (*346*) describe new developments in Cottrell electrical precipitation to reduce some of these troubles. A liquor tank replacing hoppers and the relocation of rectifiers to near furnace control has reduced cost and assured better control. Bypasses for Cottrell precipitators are discussed in a TAPPI report (*347*).

Ginodman (*348*) reported 1.487 grams total sulphur in the waste gases from the sulphate process per metric ton of pulp distributed as follows: 16% as hydrogen sulphide, 47% as methyl mercaptan, and 17% as methyl sulphide and methyl disulphide. Burning in a furnace with facilities to minimize the explosion hazard is recommended. Washing with calcium hypochloride (using the equivalent of 6 grams of chlorine per metric ton of pulp) will destroy the odors but at an excessive cost. Trobeck (*349*) oxidizes the weak black liquor before its evaporation to 50 to 55% solids and further concentration to 85 to 90%. The steam from the concentrator is contaminated and is used for preheating. Corrosion, as well as sulphur loss, is reduced. Dexter (*350*) stated that each kraft mill presents a special odor problem but the Bergstrom-Trobeck system, using the oxidation tower, multiple-effect evaporators, precipitators, burning of noncondensable gases, etc., can be coordinated to give the desired results.

Fuller and Woodside (*351*) disclose the use of sintered sodium carbonate and sulphur for make up chemical added to the green liquor. Bringman, Pascoe, and Kahn (*352*) report the results obtained in mill operation using magnesium sulphate for coagulation of the alumina and silica present in green liquor; this greatly improves causticization and settling. Reed and Gillespie (*353*) discuss centrifugal clarification of sulphate green and white liquors. Nepenin (*354*) described modern continuous recausticizing systems. The Infilco Co. (*355*) described accelerated chemical causticizing. Denisov presented a formula for ascertaining the degree of causticization (*356*).

White disclosed the mixing of lime-bearing sludge with sodium carbonate which acts as an adhesive to reduce dust (*357*). This procedure is designed for fluidized burning.

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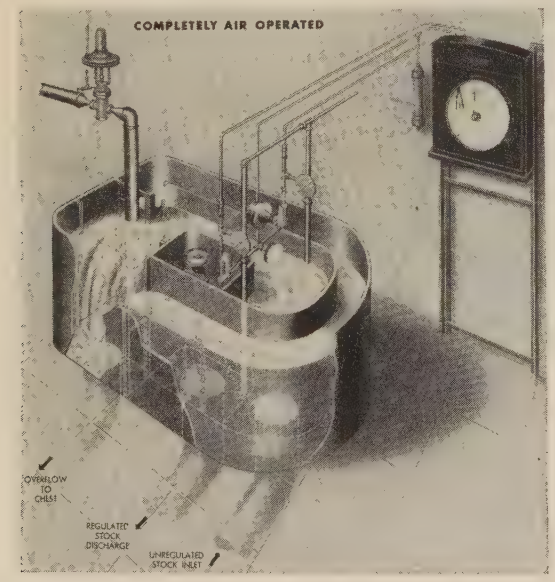

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Gibbs (*358*) discussed the themodynamics of lime kiln operation.

Whitney (*359*) presented the corrosion problems in the paper industry, von Essen (*360*) the corrosion problems in kraft pulp mills, and LaQue and Clarke (*361*) those in alkaline pulping.

Pollack, Keilen, and Drum (*362*) disclose a method for obtaining a filterable lignin from black liquor. Raff, Tomlinson, Davies and Watson (*363, 364*) discuss the use of oxidized lignin for the reinforcement of GR-S rubber. Tomlinson (*365*) discussed the use of lignin in laminates. Bennett (*366*) described the use of lignin for the fixation of ammonia.

EFFLUENTS TO STREAM AND ATMOSPHERE

Paessler and Hedgepeth (*368*), Chipman (*268*), and Winget (*367*) discussed the stream pollution problems in the southeastern United States and especially in Virginia. Observations on the toxic effect of soaps and condensate paralleled the experience reported by Lindroth (*369*) in Scandinavia. Hibbard and Veatch (*370*) disclose the use of Indulin A, B, or C (alkali lignin) for the removal of mercaptans and the possibility of their regeneration; references (*348*) and (*349*) should also be consulted. Of interest is the report by LaCoste (*371*) of black bass growing in a black liquor pond in Louisiana. Southgate (*372*) reported on sanitation and Van Horn (*373, 374*) discussed studies on the abatement of stream pollution and kraft pulping wastes in relation to aquatic environment.

PURIFICATION AND BLEACHING

Multistage chlorination and bleaching as carried on today was first put into operation in 1930 and disclosed to the public in 1932 (*375*). Since then, the equipment has been standardized and the methods of instrumental control have been perfected so that bleached pulps made by the process now comprise a substantial part of the bleached pulp (both kraft and sulphite) used in North America. Aries and Copulsky (*376*) report that production of bleached kraft pulp is now increasing more than other pulps. Casciani and Storin (*377*) disclosed the combination of chlorination followed by treatment with chlorine dioxide to high brightness but poor stability at pH less than 8 and then with peroxide at pH above 9 to high brightness and good stability. Crowley (*378*) described an installation for automatic pH control by feeding milk of lime in low density bleaching. Day and Hoos (*379*) suggest the suspension of pulp in hypochlorite and chlorite solution at pH 9 and then lowering the pH to 5 to induce the action of the chlorite. Dobson (*380*) suggested the use of ammonia and mono- and di-substituted ammonia, primary and secondary amines, amides, etc., which he claims inhibit degradation and permit the attainment of a high white at a higher viscosity. Lal (*381*) describes the Celdecor-Pomilio chlorination process. McCarthy and Hibbert (*382*) discuss multistage bleaching of kraft pulp. McEwen (*383*) and McEwen and Sheldon (*384*) disclose bleaching methods with hydrogen and alkaline peroxides, soluble phosphates, and active chlorine. McLeod (*385*) discussed the merits of sodium chlorite and chlorine dioxide for bleaching kraft and methods of preparation and application. Murphy (*386*) explained how centralized instrumentation highlights recent multistage bleach plants. Norta and Sivola (*387*) disclose a method of treatment of partially washed chlorinated pulp at a pH of less than 5 in 1 to 15% suspension for 10 to 200 minutes. Okuno (*388*) pointed out the 4 to 5% reduction in bleaching costs by using chlorine instead of hypochlorite for part of the bleaching. Russell (*389*) explained the terms and methods used in bleaching. Savage (*390*) reported a round table discussion of kraft bleaching. He also described (*391*) the new eight-stage bleach plant at Camas, Washington. Urbanski (*392*) dis-

cussed the bleaching of kraft pulp for papermaking and Wurz (*393*) reviewed alkaline processes and chlorination, together with groundwood manufacture.

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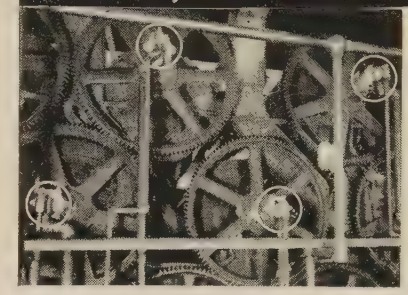

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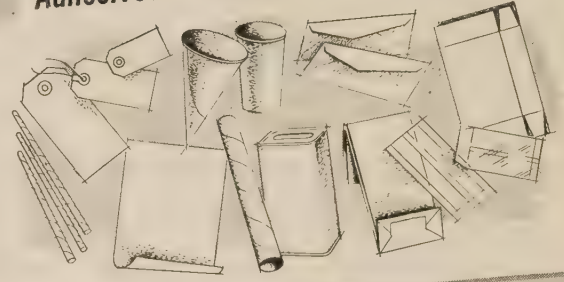

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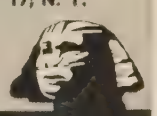

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Every spool of wire that we draw must pass three physical tests:

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Sponsored by the Alkaline Pulping Committee of the Technical Association of the Pulp and Paper Industry, Henry Vranian, Chairman.

Southern Pulp and Paper Manufacturer finances the preparation of this report annually for the Technical Association of the Pulp and Paper Industry as a service to pulp, paper, and paperboard manufacturers.

RECENT BOOKS

McGraw-Hill Directory of Chemicals and Products—1951. First Annual Ed. New York. McGraw-Hill Publishing Co. 1950. Cloth, 6 × 9, 558 pages. $20.

This book initiates a new service by the McGraw-Hill Publishing Co. It is essentially a listing of the various grades of chemical raw materials, industrial and fine chemicals, dyestuffs and their producers and manufacturers in the United States. The first 528 pages are devoted to names of products, including form, shipping label, and packing details. The remainder of the book is a listing of the producers and their various shipping points.

This book should be in the plant library of every purchasing agent. Producers of chemicals in any form should check the book to make sure that the names of their companies and products appear in the next editing. We note the absence of a number of companies that serve the pulp and paper industry.

Walden's Paper Production Year Book. 1950 Ed. New York. Walden Sons & Mott, Inc. Paper-bound, 6 × 9, 140 pages. $3.00.

This annual which is probably well known to many in the pulp and paper industry is reviewed because many more should know about it since it is packed with more useful information than meets the eye. Its compact format keeps it from being the usual formidable directory and its paper cover misleads concerning its value. On the other hand these details provide cost economy of benefit to the buyer. Although it does not describe mill equipment in much detail it does an excellent job in reporting personnel and products. It also contains a useful guide to machinery and commodity suppliers and to paper converters.

Proceedings 1949. Vol. 3. Australian Pulp and Paper Industry Technical Association, South Melbourne, Victoria, Australia. Paper-bound, $5^1/_2$ × $8^1/_2$, 530 pages. $5.00.

This is the first review of the annual proceedings of the APPITA although there have been two annual volumes issued previously. It contains the papers presented at meetings of the association and includes such subjects as: Grinding of Eucalypts, Stitching at the Press, Preventive Maintenance, Correlation of Testing Instruments, Manufacture of Paperboard, Wallboard, Soda Recovery, Pulp Characteristics Influencing Paper-Strength Properties, Odor from Kraft Mills.

Copies of all books reviewed in this issue may be obtained from the Book Dept., *Tappi*, 122 E. 42nd Street, New York 17, N. Y.

Instrumentation Studies. LVIII. A Study of Photoelectric Instruments for the Measurement of Color: Reflectance and Transmittance.

VII. The Photovolt Photoelectric Reflection Meter, Model 610

LEONARD R. DEARTH, J. A. VAN DEN AKKER, and W. A. WINK

The Photovolt photoelectric reflection meter has been investigated. This instrument has spectral characteristics which depend on the filter combination selected. It appears that it should be possible to choose a combination which would give rise to a proper effective wavelength. The intended geometry of the instrument is that of unidirectional illumination at 0° with viewing at an average angle of 45° over all azimuthal angles. The actual instrument does not accurately embody this statement, and specularly reflected light is actually included to an appreciable extent with the diffusely reflected light. The method of direct photometry is employed, in which the deflection of a sensitive galvanometer is taken as the measure of reflectance. The maximum photometrical error resulting from nonlinearity of the photocell-galvanometer system is about 2.0 points, or 4% of the galvanometer deflection. Among the difficulties encountered in the use of the instrument are extreme dependence of stability on the constancy of powerline frequency, the lack of provision for an adequate adjustment of the lamp in the search unit, and large random fluctuations that may be attributed only to poor electrical contacts in the instrument. It seems possible that most of these difficulties could be eliminated and that, after suitable calibration with reference to pulp or paper standards of material very similar to that under measurement, the instrument might be suitable for the routine control of brightness in the pulp or paper mill.

Because of the war and other circumstances beyond our control, the publication of this and several other Instrumentation Reports has been delayed several years. The study reported here of the Model 610 Photovolt reflection meter was made in 1942 and, undoubtedly, the manufacturer of the instrument has made improvements since the appearance of our report in February, 1943. However, we believe that all the report relative to principles stands as well today as it did several years ago; also, we feel that the data, although obtained on a particular instrument, should be typical of those obtained on a nonstandard "brightness tester."

During the past few years we have advocated a "special calibration procedure" where it is necessary for a mill to employ a nonstandard brightness tester. This procedure is set forth on page 11 of Report 17, Part IX to the American Paper and Pulp Association (Feb. 1, 1950).

The measurement of brightness (*1*, *8*) has become more important by reason of the war and the necessity of limiting the amount of chlorine consumed by the pulp and paper industry in the bleaching of pulp Because the demand for satisfactory instruments recently has exceeded the supply, the further inspection of photoelectric reflection meters on the Instrumentation Program has been considered desirable. It should be recalled to the reader that the several previous parts of Report 17 present the results of studies on photoelectric reflection meters (*2–6*).

Recently, there has been considerable interest in the new reflection meter manufactured by the Photovolt Corporation and designated as the Photovolt photoelectric reflection meter, Model 610.

The descriptive literature concerning the Photovolt instruments states that they are designed for "measuring the reflectance and color of solid surfaces, fabrics, pastes, powders, and opaque liquids. They are provided with a 'search unit' which is placed on the surface to be measured so that the test specimen need not be inserted into the instrument This offers the advantage that samples can be measured in rapid succession, that the sample may be of any size, and that the operator can easily select that part of the surface on which he wishes to take readings." The instrument is normally supplied with three color filters (red, green, and blue). The manufacturer is aware that readings with this instrument employing the blue filter may not accurately measure brightness as defined by Van den Akker *et al.* (*8*), but is hopeful that a proper combination of the filters would result in a measurement of reflectance that would correlate reliably with brightness. The chief aim of this report is to present data covering the use of this instrument for evaluating brightness.

Only one instrument (Serial No. 1315) was studied. However, the discussion is applicable to, and the data should be typical of, all instruments of the same model.

DESCRIPTION OF THE INSTRUMENT AND METHOD OF OPERATION

A photograph of the Model 610 Photovolt photoelectric reflection meter is presented in Fig. 1, and a schematic drawing of the lamp-photocell combination is given in Fig. 2. According to the literature covering this instrument, "Model 610 operates on 105 to 130 volts, 50 to 60 cycle a.c. current." The instrument contains a "constant voltage" transformer, which is supposed to supply a steady voltage to the lamp and thereby eliminate instability caused by variation in the line voltage. As illustrated in Fig. 2, the lamp-photo-

Leonard R. Dearth, Technical Assistant; J. A. Van den Akker, Member TAPPI; Research Associate; and W. A. Wink, Member TAPPI; Technical Associate, The Institute of Paper Chemistry, Appleton, Wis.

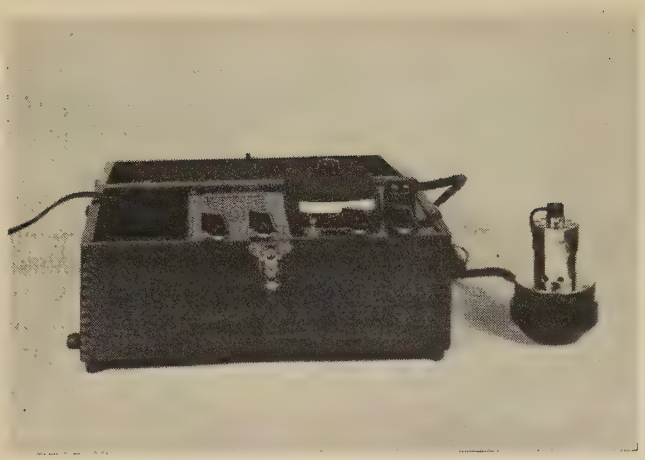

Fig. 1. The Photovolt reflection-meter

cell combination is placed on the sample (or standard) and the latter is illuminated by the collimated light from a 6-volt 15-candle power automobile headlight lamp. Reflected light falls upon a blocking-layer photocell having an annular shape. The condensed, incident light passes through the hole in this annular photocell, and it is claimed by the manufacturer that specularly reflected light returns through this aperture so that only diffusely reflected light is measured. It is stated that the angle of incident light is 0° with the normal, that the average angle of the reflected light is approximately 45° and, hence, "the results obtained are very nearly the same as for 45° angle of incidence and 0° viewing angle." The photocurrent is read directly by the galvanometer of the instrument, the deflection of which is taken as the measure of reflectance.

Situated immediately over the measuring photocell and arranged back-to-back with the measuring photocell is a so-called "compensating photocell," the purpose of which is to enable the operator to adjust the instrument to zero deflection for a minimum reflectance standard of any desired value. This second photocell is not so connected in the circuit as to eliminate the influences of variations in line voltage in the usual way, but brings about a steady zero deflection in spite of line voltage fluctuations. The use of the "suppressed zero" enables the operator to expand the interval between any two standards to the full scale of the galvanometer. The compensating photocell supplies a current (excited by the light from the lamp) which, through the use of the two left-hand knobs (rheostats), enables the operator to obtain the suppressed zero when the search unit is placed upon the darker standard. Alternatively, "when these knobs are turned all the way clockwise, the zero point of the galvanometer scale indicates zero reflectance." (The galvanometer had been adjusted previously to mechanical zero with the instrument turned off.) Measurements may be made with fairly satisfactory reproducibility only after the instrument has "warmed up" over a period of about half an hour.

The procedure, which is apparently preferred by the manufacturer, involves the use of the suppressed zero—that is, setting the instrument upon zero for a dark standard of known reflectance not zero—followed by setting the galvanometer deflection to 100 when the search unit is placed on a "light standard" of known reflectance not equal to 100%; the reflectance of the sample is calculated subsequently by means of a formula which involves the reflectance of the dark standard, the reflectance of the light standard, and the galvanometer readings obtained when the search unit was placed on the sample and the standards. The dark standard need not have low reflectance; as an illustration, it may be 60% reflectance where it is desired to expand the 60 to 80% range to that of 0 to 100 galvanometer divisions. The advantage of an expanded scale is illusory, and it has been taken for granted that in typical routine work, where it is desired to obtain readings fairly rapidly, the preferred procedure would be to operate the instrument after adjusting the galvanometer deflections to the values numerically corresponding to the reflectances of the black and white standards. Moreover, the suppressed zero cannot be effectively used whenever optically dense filters (such as those required in the measurement of brightness) must be employed. The reason for this is that the use of the suppressed zero requires considerable reserve sensitivity, which is lacking when dense filters reduce the amount of light incident on the photocell.

The procedure followed in the study described in this report was to employ a dark standard comprising a black body having essentially zero reflectance, and a white standard having a reflectance close to 100% on the magnesium oxide scale. In this case, the Photovolt equation for the reflectance reduces simply to the statement that the reflectance of the sample is numerically equal to the galvanometer reading.

In order that the measured reflectance may be of significance, it is necessary that a proper filter or filter combination be employed.

SPECTRAL CHARACTERISTICS OF THE INSTRUMENT

The optical design of this instrument is such that sensitivity is adequate for the use of infrared-free light filtered by means of a blue stenochromatic filter.

It was recognized by both the manufacturer and the Institute that the blue-glass filter with which the instrument is equipped would probably not be satisfactory for the measurement of a blue reflectance correlating with brightness. The spectral transmission of the blue-glass filter runs to high values at wavelengths near 400 mmu, and is generally very different from that required to give the proper combination of spectral transmission and spectral response of the photocell (*1*). To demonstrate the correctness of this consideration, the brightness of a series of papers were determined with the blue-glass filter as well as with a more proper combination of filters. The reflectances obtained with the blue-glass filter, together with the individual deviations from the known brightnesses, are presented in the third column of Table I. The series of samples used in this study included bleached and unbleached kraft and sulphite pulp, and covered a brightness range from 22 to 83%. Inspection of the results obtained using the blue-glass filter will reveal that the individual deviation varies from −1.3 to −8.8%. Obviously, a spread of discrepancies of this order of magnitude will not permit reliable correlation between instrumental readings and brightness. The large discrepancies, of course, are to be attributed to the fact that the blue filter is not suitable.

A more appropriate combination of a Wratten 49 filter and Jena BG-18 filter was next attempted. The first Wratten 49 filter was soon destroyed by the high

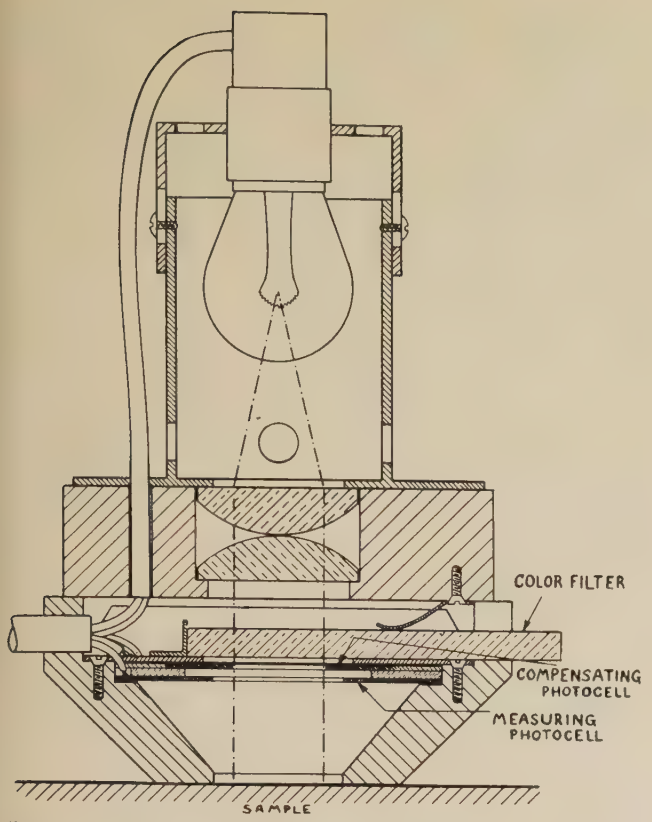

Fig. 2. Search unit of Photovolt photoelectric reflection meter

temperatures obtained in the search unit. (In order to secure adequate sensitivity with stenochromatic filters, a fairly intense lamp must be employed and the search unit becomes heated to a high temperature.) However, by resorting to a special method of handling the Wratten filter, it was found possible to operate over a sufficiently long period of time to obtain the results presented herein. The data obtained with the more correct filter combination are given in the fifth column of Table I. It will be observed that the discrepancies are much smaller, covering a range from −1.2 to +2.1%, a total spread of approximately 3.3%. This order of variation is several times too large for the instrument to be satisfactory for the general measurement of brightness; however, were it not for other defects discussed below, a total spread of approximately 3 points would indicate that the instrument might be satisfactory for the routine control of the brightness of a given grade of paper made under constant papermaking conditions from a given furnish.

The effective wavelength of the instrument was determined by the preferred spectrophotometric method involving the measurement of transmittance of a "linear filter" (*1, 8*). Because the instrument cannot be employed to measure transmittance, except by an arrangement involving a double passage of the light through the linear filter (thereby giving a result which is a function of the square of the transmittance), a special arrangement was devised. This involved an auxiliary 50-candle power lamp placed about 4 inches from the sample aperture of the search unit. The aperture was covered with the Wratten 49 and Jena BG-18 filters and a piece of flashed glass which was known from previous investigations to diffuse light almost perfectly; the flashed surface of the diffusing glass was in immediate contact with the aperture

Two similar optical cells were filled, respectively, with distilled water and a solution having a linear spectral transmittance (*1*). First, the cell containing distilled water was placed in the light beam between the lamp and sample aperture, and the instrument was adjusted to give an appreciable galvanometer deflection. The cell containing the distilled water was then replaced by the cell containing the solution, and the galvanometer reading was noted. Upon correcting the galvanometer deflection for nonlinearity (see below), the transmittance was calculated. By the use of the G. E. recording spectrophotometer, it was then established that the effective wavelength was 464 mmu, or about 7 mmu higher than the standard value (*1*).

The greater effective wavelength of the Photovolt reflection meter is undoubtedly to be attributed to the fact that the relative spectral response of the photocell used is of entirely different slope from that which is commonly employed in the measurement of brightness. It should be possible to find a combination of filters which would give rise to the standard effective wavelength, namely, 457 mmu. It may be, however, that there is some tendency for the spectral discrepancy to compensate for deviations arising in geometrical and photometrical characteristics; hence, attention should be directed to the latter deviations, as well as to the spectral error.

GEOMETRICAL AND PHOTOMETRICAL FEATURES

As shown in Fig. 2, the light from the incandescent lamp is collimated by a pair of simple lenses. However, the geometry is not well defined because of the fact that the filament of the lamp employed is rather extensive in comparison with the physical dimensions of the search unit. In consequence, the rays of the condensed light cover a substantial range of angles centering about the normal to the sample. In the advertising literature for this instrument, it is stated that "this search unit registers dispersed [diffuse] reflection only, inasmuch as specularly reflected light returns through the aperture of the photocell." Although it is true that a large portion of the specularly reflected light returns through the aperture in the photocell, special measurements per-

Table I. Comparison of Blue Reflectances with Standard Brightness

Sample	*Standard brightness, %*	*Photovolt reflectance: glass filter* %	*Difference*	*Photovolt reflectance: Wratten 49 and Jena BG-18 filters* %	*Difference*
19	22.4	21.0	−1.4	22.4	0.0
13	22.8	21.5	−1.3	23.7	+0.9
6	25.9	21.5	−4.4	26.0	+0.1
5	26.3	23.1	−3.2	26.0	−0.3
4	30.5	27.8	−2.7	29.6	−0.9
7	36.9	31.3	−5.6	37.1	+0.2
11	37.2	34.1	−3.1	38.2	+1.0
22	50.4	42.4	−8.0	49.4	−1.0
30	52.7	45.0	−7.7	53.0	+0.3
20	54.0	45.3	−8.7	52.8	−1.2
18	55.3	50.3	−5.0	56.4	+1.1
29	60.3	52.8	−7.5	60.6	+0.3
27	62.8	54.0	−8.8	62.3	−0.5
8	63.5	58.0	−5.5	63.3	−0.2
26	65.3	57.8	−7.5	65.6	+0.3
3	66.0	61.0	−5.0	65.9	−0.1
9	67.5	64.9	−2.6	69.6	+2.1
10	68.1	63.9	−4.2	69.8	+1.8
28	70.1	63.5	−6.6	69.7	−0.4
C	73.7	69.7	−4.0	74.0	+0.3
24	73.7	68.1	−5.6	74.1	+0.4
1	76.3	72.9	−3.4	76.0	−0.3
D	82.9	78.3	−4.6	82.3	−0.6

formed with a front surfaced mirror of high optical quality failed to verify this assertion. With the instrument adjusted at zero with a black cavity of essentially zero reflectance and at 100% on the magnesium oxide scale, the apparent reflectance of the front surfaced mirror was 17.5%. This specular component of the reflected light causes noticeable deviations from an instrumental scale based on diffuse reflection. Such deviations depend upon the surface characteristics of the samples. For example, inspection of the samples employed in this study reveals that the Photovolt reflection meter tends to read high on glossy samples and low on rough samples.

Discrepancies resulting from specular reflection cannot be more than 1 or 2 points. In a mill where the finish of the paper is well controlled, a correlation chart for each particular paper would undoubtedly take care of this source of geometrical error. Note should be made of the possibility that this source of error could be relatively large in the case of black or dark gray samples.

The annular photocell is so arranged that the average angle of accepted rays is approximately 45° for all azimuthal angles. Thus, this instrument measures a reflectance which is independent of sample orientation. This is considered by some as a good feature in control work; it should be recalled, however, that in a normal viewing with the eye the relative reflectance of a specimen will depend on orientation. Moreover, the normal brightness scale requires that the machine direction of the paper be oriented parallel to the optical plane.

This instrument is to be classified as dependent on the method of direct photometry, in which the deflection of a galvanometer is taken as the measure of reflectance. As indicated in previous Instrumentation Reports, this method is subject to an appreciable error because of the nonlinearity of response of the photocell-galvanometer combination, unless the galvanometer resistance and/or the intensity of light is very low.

Working with a Wratten 49 filter, and using a Jena BG-18 filter to eliminate the infrared light, it was found that a lamp of reasonably high power had to be placed too close to the search unit to permit an accurate check of linearity by means of the inverse-square law. Hence, the scale was checked at one point by the use of a standard screen having a known transmission. With the instrument set up with a flashed diffusing glass and filters, as discussed above for the measurement of effective wavelength, the transmission of the standard screen was measured.

Under the conditions of illumination employed in the determination of effective wavelength (intensity of light such that, with no screen placed in the beam, the deflection was approximately midscale), the measured transmission of the standard screen was 42.6%. For the given angular spread of light rays, the transmission of the standard screen is 42.3%. Hence, the correction was relatively small, being +0.3 point.

In normal use, however, the deflection of the galvanometer corresponding to maximum light intensity is close to full scale. It is well known that photocells of the type used in this instrument show greater deviations from linearity at higher light levels. Therefore, the light level was increased so that the maximum deflection was close to full scale (100 divisions). Under these circumstances, the measured transmission of the standard screen was 44.0%, whereas the known transmission

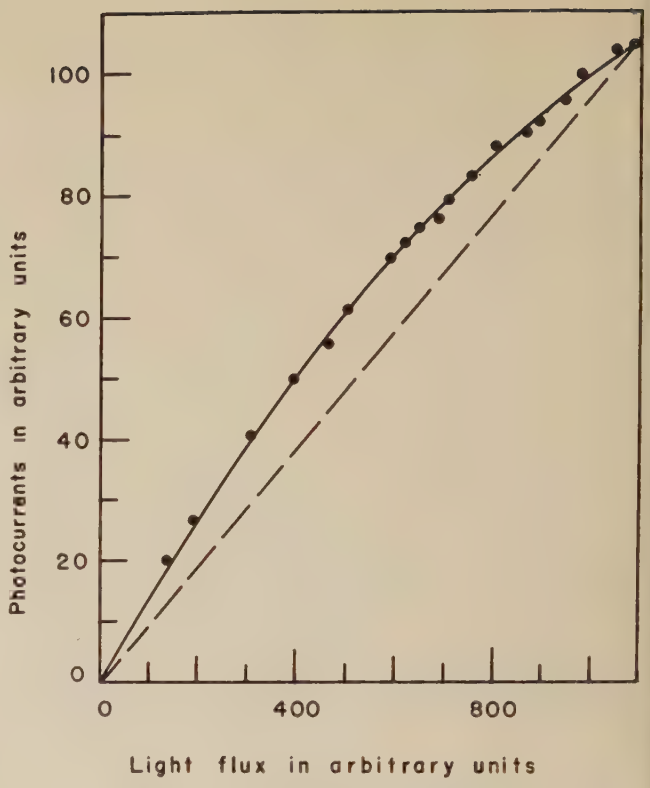

Fig. 3. Photocurrent vs. light flux

under the new range of angles was 42.0%. Hence, the discrepancy under the normal conditions of operation, for readings near midscale (where deviations from perfect linearity are maximum), is about 2.0 points, or 4% of the actual deflection. The form of the photometric error curve would be very similar to that shown in Fig. 3, obtained in the study of another instrument employing a blocking-layer photocell. Although the photometric error is very significant wherever absolute reflectance must be determined, it is, without doubt, reasonably consistent, so that proper calibration would take it into account.

GENERAL FEATURES OF THE INSTRUMENT

An unfortunate characteristic of the Photovolt reflection meter, which might be eliminated through the proper use of storage batteries, is the sensitivity of the device to changes in frequency of the power line. A "constant voltage" transformer furnishes power to the lamp. The transformer employed is presumably like most such devices, using the method of operating at high magnetic flux density in the core of the transformer, in a circuit adjusted to be near electrical resonance. In any case, the intensity of light from the lamp, as measured by the deflection of the galvanometer, varies with frequency in an extremely marked manner, as shown by the data in Table II.

These data show that, for a frequency change from 55 to 65 cycles per second, the relative intensity of the lamp increases by about 470%. Even on a relatively well controlled line, where the frequency is maintained within approximately ±1 cycle per second, fluctuations of at least ±15% may be expected. Fluctuations of this order of magnitude are unsatisfactorily large in view of the fact that ordinary colorimetric work requires a precision of measurement (reproducibility) of the order of 0.3%, or better. In many mills supplying their own power from lines not connected to larger,

Table II. Dependence of Instrumental Response on Power-Line Frequency[a]

Frequency, cycles/sec.	*Galvanometer deflection, divisions*
45	3
50	8
55	19
56	22
57	26
58	30
59	35
60	40
61	47
62	53
63	59
64	64
65	90

[a] Obtained with variable-speed, sine-wave alternator.

well-regulated systems, frequency variations often amount to several cycles per second. It is obvious that a constant voltage transformer could not be used under these circumstances. It is regrettable that the manufacturers of the common type of constant voltage transformers do not place more emphasis upon the frequency sensitivity of these devices.

It is difficult to adjust the lamp in any direction other than up or down, so that centering of the beam of light in the sample aperture is not simple. The accuracy of the measurement of reflectance is dependent, in part, upon the scattering of light in translucent material, which has been termed "irradiation effect" and "translucency effect" (*7, 8*); it is necessary, therefore, that one be able to adjust the position of the light beam and the size of the spot of light in the sample aperture. The errors resulting from the translucency effect are relatively small when the standard and sample are the same (or nearly the same) kind of paper or pulp. Where a reflection meter such as that under discussion must be calibrated through the use of standards of material the same as that under measurement, the use of standards which are composed of such materials as pot opal glass, tile, enamel, etc. is not recommended. Standards composed of the latter materials may be employed if they are evaluated relative to paper standards of a known brightness and characteristic the same (or nearly the same) as the samples to be measured, although the procedure is risky if the working standards are not frequently checked.

The electrical contacts in the instruments are very poor. In view of the fact that they can be improved, this statement should not be interpreted as a major criticism. However, fluctuations that are clearly due to poor electrical contacts are large and very bothersome. When this instrument has been in operation for a few hours and is therefore in thermal equilibrium—even when the frequency of the power line is very constant—there are random, erratic fluctuations of large magnitude which can be attributed only to poor electrical contacts; occasionally these larger fluctuations may be caused by a slight jarring of the instrument or by a general vibration of the building.

The high temperature of the search unit eliminates the possibility of using gelatin filters over any appreciable period of time. It will be necessary to employ heat-resisting glass filters of suitable spectral transmission or to modify the design of the search unit.

Experiments with a black cavity showed that discrepancies occasioned by stray light within the search unit are negligible.

It is felt that improvements indicated in this report might lead to an instrument which, when suitably calibrated against samples of paper or pulp similar to those measured, would be satisfactory for the routine control of brightness in the pulp or paper mill.

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4. Report No. 17 to APPA. A study of photoelectric instruments for the measurement of color: reflectance and transmittance. Part IV. The Hunter multipurpose reflectometer. Oct. 20, 1937. Instrumentation Studies XXIX. *Paper Trade J.* 107, no. 25: 29–40 (Dec. 22, 1938).
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7. Report No. 8 to APPA. Adaptability of the G. E. reflection meter as a color analyzer. Parts I–III. Nov. 20, 1935, Dec. 18, 1936. Instrumentation Studies XIII. *Paper Trade J.* 104, no. 18: 47–53; no. 19: 51–63; no. 20: 45–49 (May 6, 13, 20, 1937).
8. Van den Akker, J. A., Nolan, Philip, and Wink, Willmer A. The physical basis of standardization of brightness measurement. *Paper Trade J.* 114, no. 5: 34–40 (Jan. 29, 1942); *Tech. Assoc. Papers* 25: 263–269 (June, 1942).

This report is one of a series issued as a part of the Instrumentation Program of the American Paper and Pulp Association. The data in this paper were reported to the Association on Feb. 8, 1943.

EMPLOYMENT SERVICE

Positions Wanted

E90-50. Converting Chemist. Fifteen years' experience in paper manufacturing and converting, including gumming, waxing, and printing. Desires position with a progressive company in production or sales.

E91-50. Chemical Engineer. B.S. 27, married. Experienced in paper quality control, including statistical methods. Desires similar position or that of pulp or paper mill chemist or engineer.

E92-50. Supervisor of production or quality control. At present located in Canada but desires to locate in the United States. Five years experience in a modern kraft mill in studies of paper control, sulphate pulp, and bleaching. Has had some sales service experience.

E93-50. Mill affiliation sought by young man in a technical-sales position. Has operated technical service department of a large paper distributor for the past three years. Through knowledge of paper, its properties, and uses. Age 25. Married. Attended universities in the U.S.A. and Europe.

E94-50. Graduate Chemical Engineer with 25 years' pulp and paper experience, research, development, sales service, plant management, and quality control—desires responsible position with progressive concern where initiative and competence are desired.

Positions Open

P85-50. Shipping superintendent. To warehouse and ship paper bags, rolls, sheets, and miscellaneous paper products. Must be able to direct labor efficiently and economically. Large kraft pulp and paper mill located in Southeast. Climate excellent. State full qualifications, experience, education, and references.

UNITED STATES PATENTS ON PAPERMAKING

First Quarter, 1950 — *Compiled by CLARENCE J. WEST*

The following list of patents has been compiled from the current numbers of the Official Gazette of the United States Patent Office. Because, as a rule, only one claim is published in the Gazette, it is not claimed that the list is complete; also, it is possible that the list may contain some patents that do not apply specifically to pulp and paper manufacture.

Copies of any of the following patents can be obtained from the United States Patent Office, Washington 25, D. C., by sending twenty-five cents for each patent desired.

January 3, 1950

2,492,968. Web severing and clamping means. Otha Cooksey, assignor to The Collmar Corporation, Chicago, Ill. Filed June 3, 1946. 15 claims. (Cl. 164–39.) A web-severing device for coating machines is actuated by a solenoid; wrap-up, which may result from cutting the web too far from the rolls, is prevented.

2,492,974. Nozzle member used for the drying of textile and other materials. Julien Dungler, Basel, Switzerland. Filed April 30, 1947. 1 claim. (Cl. 34–156.) A paper or textile drier is provided with hot-air nozzles which are arranged so that the air streams are directed at acute and right angles with the surface of the product.

2,493,031. Prevention of degradation of textile fibers by acids. John D. Reid, Wesley K. Ward, Jr., and Laurence W. Mazzeno, Jr., assignors to the United States of America as represented by the Secretary of Agriculture. Filed March 29, 1946. 1 claim. (Cl. 206–84.) A multi-ply paper bag for acid fertilizer is sewed with thread which has been impregnated with about 20% by weight of triethanolamine.

2,493,086. Snap-on-cover. James D. Reifsnyder, assignor to Lily-Tulip Cup Corporation, New York, N. Y. Filed Feb. 19, 1949. 5 claims. (Cl. 229–43.) A snap-on paper cover is provided for ice cream containers having a beaded upper lip.

2,493,176. Carton blank. Earl B. Whitehead, assignor to Atlantic Carton Corporation, Norwich, Conn. Filed Jan. 9, 1948. 2 claims. (Cl. 229–33.) The cover of a preformed paper carton is provided with locking tabs for holding the cover closed.

2,493,189. Carton. William P. Frankenstein, Cincinnati, Ohio. Filed Aug. 20, 1945. 10 claims. (Cl. 229–34.) A hollow-walled knocked-down carton is provided with fold or score lines which may be broken prior to setting up.

2,493,194. Manufacture of bonded fiber mats. Arne W. Heine, assignor to Wood Conversion Company, St. Paul, Minn. Filed Nov. 29, 1946. 15 claims. (Cl. 154–29.) A mechanism is described for transferring an undried, bonded, low-density fiber mat from a forming to a drying belt.

2,493,243. Method and apparatus for forming cellular structures. Joseph F. Godwin, Salem, N. J. Filed March 26, 1946. 12 claims. (Cl. 93–37.) A method and apparatus are provided for assembling slotted corrugated board partitions to form a cellular unit.

2,493,281. Folding paper box. Wilbur G. Anderson, Jr., assignor to American Box Board Company, Grand Rapids, Mich. Filed Nov. 24, 1947. 7 claims. (Cl. 229–23.) The upper edge of a reusable paperboard container for baked goods is reinforced with an open wire frame.

2,493,337. Lined carton or container. Kenneth T. Buttery, assignor to Sutherland Paper Company, Kalamazoo, Mich. Filed Oct. 25, 1945. 5 claims. (Cl. 229–14.) A moistureproof or waterproof liner is adhesively secured to the walls of a container so that the liner is opened or erected when the walls are erected.

2,493,338. Covered box or container. Kenneth T. Buttery, assignor to Sutherland Paper Company, Kalamazoo, Mich. Filed April 16, 1947. 10 claims. (Cl. 229–36.) The body end walls of a covered container are provided with diagonal weakening lines which form joint flaps at the front ends of the walls.

2,493,348. Bag with extensible sealed liner. Edgar B. Hoppe, assignor to St. Regis Paper Company, New York, N. Y. Filed Sept. 21, 1944. 1 claim. (Cl. 229–55.) A method of sealing a multi-ply bag.

2,493,349. Bag with extensile sealed liner and method for making same. Edgar B. Hoppe assignor to St. Regis Paper Company, New York, N. Y. Filed Nov. 30, 1946. 2 claims. (Cl. 229–55.) A method of sealing a multi-ply bag.

2,493,410. Apparatus for folding paper sheets. Edgar J. La Cour, assignor to Orchard Paper Company, St. Louis, Mo. Filed Sept. 7, 1945. 2 claims. (Cl. 270–61.) The tangential rolls of a paper folding device incorporates sections of highly frictional material on their peripheries.

2,493,486. Tall oil esters. Sylvan Owen Greenlee, assignor to Devoe & Raynolds Company, Inc., Louisville, Ky. Filed April 10, 1946. 4 claims. (Cl. 260–18.) A synthetic drying composition consists of tall oil esters of a high molecular weight, resinous, polymeric polyhydric alcohol, which is a polyether derivative of a dihydric phenol.

2,493,562. Loading pallet. Paul J. Yarman, assignor to The Ohio Boxboard Company, Rittman, Ohio. Filed April 4, 1947. 5 claims. (Cl. 248–120.) A loading pallet incorporates a double-faced corrugated board platform which is supported by compact paperboard spacer blocks.

2,493,583. Method of filling containers and apparatus therefor. Henry A. Johnson, Kansas City, Mo. Filed April 27, 1946. 3 claims. (Cl. 18–55.) A method and apparatus for holding and reinforcing collapsible paperboard containers while filling with a fluid and allowing the fluid to solidify or freeze.

2,493,590. Apparatus for positioning cores. William G. Mueller, Karl Klause, and Anton M. Meier, assignors to Marathon Corporation, Rothschild, Wis. Filed Oct. 22, 1947. 15 claims. (Cl. 29–211.) A device is provided for accurately positioning a short tubular core on a rewinder shaft.

2,493,604. Insulating paper of asbestos and bentonite. Theodore R. Walters, assignor to General Electric Company, Schenectady, N. Y. Filed Nov. 6, 1944. 2 claims. (Cl. 92–3.) The paper contains 5 to 35 parts of bentonite and 65 to 95 parts of asbestos fiber.

2,493,671. Container closure. Raymond H. Horning, assignor to Oswego Falls Corporation, Fulton, N. Y. Filed Dec. 4, 1946. 5 claims. (Cl. 229–5.5.) The flanged closure of a paperboard milk container is coated with a thermoplastic adhesive for sealing the flange to the body of the container.

January 10, 1950

2,493,798. Polychloroprene and preparation thereof. Henry H. Abernathy and George W. Scott, assignors to E. I. du Pont de Nemours & Company, Wilmington, Del. Filed July 8, 1947. 6 claims. (Cl. 260–27.) A process is given for the preparation of stable polychloroprene latices which can be used for impregnating paper.

2,493,866. Recovery of abietic acid. Frederick H. Gayer, assignor to Continental Research Corp., Chicago Heights, Ill. Filed May 3, 1947. 23 claims. (Cl. 260–97.6.) The tall oil resin acids are dissolved in ammonium hydroxide, the ammonia is volatilized, and abietic acid is crystallized from the dispersion.

2,493,867. Recovery of abietic acid in the form of its quarter salt. Frederick H. Gayer, assignor to Continental Research Corp., Chicago Heights, Ill. Filed May 3, 1947. 21 claims. (Cl. 260–97.6.) Tall oil is distilled at 13 mm. until about 45% remains; the residue is neutralized with sodium hydroxide and about 65% of the resin acid soap is decomposed with sulphuric acid; the quarter salt of abietic acid separates.

2,493,968. Method and apparatus for making batt-covered sheets, Charles Hepner, Chicago, Ill. Filed Oct. 17, 1946. 19 claims. (Cl. 154–31.) This covers a method for making metal wool-covered paper-backed pads.

2,494,075. Paper jogger. Carl S. Weyandt, Homer City, Pa. Filed March 18, 1946. 3 claims. (Cl. 271–89.) The horizontal deck of a paper jogger vibrates in such a manner that two edges of a stack of paper are jogged simultaneously.

2,494,098. Automatically controlled recovery of heat and SO_2 gas from relief gases and waste liquor. Carl J. Lockman, assignor to American Heat Reclaiming Corporation, New York, N. Y. Filed June 22, 1943. 1 claim. (Cl. 92–11.) The relief gases and the flash vapor from the liquor are passed to a common chamber and the resulting mixture is transferred to a heat-exchange system.

2,494,151. Powder box. James L. Younghusband, Chicago, Ill. Filed Dec. 6, 1947. 1 claim. (Cl. 229–43.)

2,494,176. Adhesive applying mechanism. Stanley R. Howard, assignor to Pneumatic Scale Corporation, Limited,

Quincy, Mass. Filed Aug. 22, 1947. 4 claims. (Cl. 91–12.) A mechanism controls the quantity of adhesive transferred to a continuously moving web of bag-forming material.

2,494,177. Package and method of making the same. Oscar H. Hultin, assignor to Pneumatic Scale Corporation, Limited, Quincy, Mass. Filed March 16, 1946. 1 claim. (Cl. 206–45.34.) A display package consists of a rectangular cellophane bag overwrapped with a strip of cardboard.

2,494,186. Sheet feeding mechanism. Joseph Magnusson, assignor to Pneumatic Scale Corporation, Limited, Quincy, Mass. Filed June 4, 1947. 11 claims. (Cl. 271–32.) A labeling machine.

2,494,234. Carton. William P. Frankenstein, Cincinnati, Ohio. Filed Nov. 29, 1946. 15 claims. (Cl. 229–34.) A hollow-walled carton embodies a step or shoulder portion either inside or outside the carton.

2,494,245. Wire guide for papermaking machines. Karl A. Karlsson, Skapafors, Sweden. Filed April 8, 1946. 6 claims. (Cl. 74–241.) A wire guide includes a pendulum system which acts to restore the wire to its normal position.

2,494,334. Process of and apparatus for making longitudinally and laterally stretchable creped webs. Paul W. Dorst assignor to Cincinnati Industries, Inc., Lockland, Ohio. Filed July 29, 1946. 17 claims. (Cl. 154–30.) A method is claimed for controlling the degree of stretchability imparted to a creped paper web.

2,494,431. Continuous corrugating mechanism. Karl J. Eckstein, Hamilton, Ohio. Filed June 28, 1945. 15 claims. (Cl. 154–30.)

2,494,437. Process of manufacturing collapsible cellular container partitions. Walter C. George and Arthur P. Klasing, assignors to Gaylord Container Corporation, St. Louis, Mo. Filed July 30, 1948. 6 claims. (Cl. 93–37.) A collapsible 24-cell container partition is formed from eight webs of board.

2,494,446. Slide dispensing package with reinforced wrapper. George A. Moors, assignor to National Biscuit Company, New York, N. Y. Filed Sept. 21, 1944. 7 claims. (Cl. 206–45.33.) A package for bakery products.

January 17, 1950

2,494,542. Method of bleaching pulp. Ferri Casciani and Gordon K. Storin, assignors to Niagara Alkali Company, New York, N. Y. Filed May 2, 1946. 6 claims. (Cl. 8–106.) Kraft pulp is bleached with chlorine or a hypochlorite, then with chlorine dioxide, and finally with a peroxide.

2,494,545. Lignin resins and process of making same. Raymond N. Evans and Angelo P. Ingrassia, assignor to Masonite Corporation, Laurel, Miss. Filed April 20, 1946. 6 claims. (Cl. 260–17.5.) A water-resistant board is prepared from an acid-hydrolyzed lignocellulose and a resin from lignin and polymerized furfuryl alcohol.

2,494,559. Coating and impregnating composition. Louis J. Jubanowsky, assignor to The Baker Castor Oil Company, Jersey City, N. J. Filed Aug. 17, 1948. 11 claims. (Cl. 106–250.) A fusible impregnating and coating composition consists of an ester melting at 50° or above and a nonhydrogenated air-blown polyhydric alcohol ester of an unsaturated fatty acid.

2,494,631. Pulp decoy. Arthur H. Risch, assignor to Pulp Reproduction Company, Milwaukee, Wis. Filed Sept. 5, 1946. 3 claims. (Cl. 43–3.) A molded pulp product.

2,494,708. Stable bituminous emulsion and preparation thereof. Donald C. Jesseph, Los Angeles, Calif. Filed July 27, 1946. 4 claims. (Cl. 252–311.5.) An emulsion of bitumen and water is stabilized with ammonium lignosulphonate.

2,494,723. Creping corrugated papers. William W. Rowe, assignor to Cincinnati Industries, Inc., Lockland, Ohio. Filed Feb. 8, 1947. 8 claims. (Cl. 154–30.) The apparatus is designed to crepe and corrugate paper in one operation.

2,494,730. Palletized container. Arthur H. Thursby, assignor to Inland Container Corporation, Indianapolis, Ind. Filed Jan. 29, 1949. 5 claims. (Cl. 229–16.) A corrugated board palletized container is essentially an open box with filled corrugated cleats secured to the bottom of the box.

2,494,743. Manufacturing pulp articles. Merle P. Chaplin, assignor to Chaplin Corporation, Portland, Maine. Filed April 30, 1942. 4 claims. (Cl. 92–56.) A method of forming molded pulp articles.

2,494,744. Glue. Moss B. Christian and Horace F. Ladd, assignors to Chicago Mill and Lumber Company, Chicago, Ill. Filed Feb. 6, 1948. 6 claims. (Cl. 106–133.) A glue for paper and wood veneer consists of soya bean flour, sodium hydroxide, and a resin from southern longleaf pine.

2,494,905. Method and apparatus for heat sealing. Harold F. Shumann, Pittsburgh, Pa. Filed Sept. 11, 1946. 6 claims. (Cl. 93–20.) A method of selectively heat sealing less than all of a plurality of superimposed plies of flexible materials.

2,494,965. Composite commodity wrapper. Shy Rosen, assignor to Milprint, Inc., Milwaukee, Wis. Filed Dec. 27, 1946. 2 claims. (Cl. 229–87.) A flexible wrapper for sliced bread.

2,495,003. Taping machine. Frank J. Haas, assignor to Boxart Machine Company, Brooklyn, N. Y. Filed June 3, 1947. 7 claims. (Cl. 93–56.) The machine is for taping the corner joints of folded paper boxes.

2,495,008. Adhesive tape. Clark M. Keaton, assignor to American Marietta Company, Seattle, Wash. Filed Jan. 18, 1947. 3 claims. (Cl. 117–68.5.) The adhesive consists of a polyamide resin, Polypale ester No. 1, and paraffin wax.

2,495,043. Laminated product and process of making same. Grant S. Willey and Kenneth S. Ruthman, assignors to United States Gypsum Company, Chicago, Ill. Filed Oct. 8, 1943. 21 claims. (Cl. 154–133.) A hardboard about 0.064-inch thick is formed from individual sheets of paper containing ferric sulphate.

2,495,066. Dusting and polishing paper. Hilton I. Jones, assignor to The Munising Paper Company, Munising, Mich. Filed Dec. 12, 1945. 17 claims. (Cl. 15–208).

2,495,148. Method of manufacturing continuous electrodes. Ragnar Tanberg, Drammen, Norway. Filed July 17, 1946. 3 claims. (Cl. 13–18.) Evaporated sulphite waste liquor is used as the binding agent.

January 24, 1950

2,495,248. Smelting process of recovering chemicals from the black liquor derived in pulp mills. Emilio A. J. Gagliardi, Charles Mellor, and Alexander L. Hamm; assignors to Combustion Engineering-Superheater, Inc., New York, N. Y. Filed Aug. 8, 1946. 3 claims. (Cl. 23–48.) The sulphidity of the white liquor from the smelt of the black liquor is controlled by substantially continuously coke particles into the black liquor before combustion.

2,495,285. Wax and polymeric polyketone compositions and articles coated therewith. Harvey H. Hoehn, assignor to E. I. du Pont de Nemours & Company, Wilmington, Del. Filed March 25, 1949. 13 claims. (Cl. 260–28.5.) A polymeric polyketone (from ethylene and carbon monoxide), polyisobutylene, and paraffin wax are used as a coating for labels.

2,495,575. Methods and materials for stiffening paper match splints. Herbert Ellern, assignor to Universal Match Corporation, St. Louis, Mo. Filed March 7, 1947. 1 claim. (Cl. 52–26.) A stiffening impregnant for boards contains white paraffin, limed rosin, opal wax, and carnauba wax.

2,495,640. Method of molding. Irving E. Muskat, Plainfield, N. J., assignor to Marco Chemicals, Inc. Filed June 8, 1945. 17 claims. (Cl. 154–110.) A method is described for molding laminated resin sheets at low pressure.

2,495,656. Pulpstone. George N. Jeppson and Wallace L. Howe, assignors to Norton Company, Worcester, Mass. Filed Aug. 27, 1947. 4 claims. (Cl. 51–206.4.) Very small abrasive blocks are used to reduce the internal heat stresses which tend to rupture larger blocks or segments.

2,495,676. Display and shipping container. William J. Wells, assignor to F. N. Burt Company, Inc., Buffalo, N. Y. Filed Jan. 22, 1945. 3 claims. (Cl. 206–44.) A cardboard shipping container for cigarettes is designed for display purposes.

January 31, 1950

2,495,720. Hunter's decoy. Roy D. Heymann, assignor to Animal Trap Company of America, Lititz, Pa. Filed April 5, 1945. 1 claim. (Cl. 43–3.) A molded pulp product.

2,495,721. Hunter's decoy. Roy D. Heymann, assignor to Animal Trap Company of America, Lititz, Pa. Filed March 18, 1947. 17 claims. (Cl. 43–3.) A molded pulp product.

2,495,807. Double walled box or carton. Kenneth T. Buttery, assignor to Sutherland Paper Company, Kalamazoo, Mich. Filed Sept. 9, 1946. 6 claims. (Cl. 229–34.) A collapsible double-walled meat carton.

2,495,994. Upright folding machine. Robert N. Ward and William H. Matthews, assignors to The Egry Register Company, Dayton, Ohio. Filed Nov. 11, 1944. 4 claims. (Cl. 270–79.) A machine for fan folding a continuous paper web.

2,496,043. Leakproof package and method of making same. Robert A. Farrell, assignor to Marathon Corporation, Rothschild, Wis. Filed July 26, 1946. 13 claims. (Cl. 2295.5.) A method of sealing a tubular leakproof package is described.

2,496,050. Froth flotation of iron ores, including use of alkali phosphate. Earl C. Herkenhoff, assignor to American Cyanamid Company, New York, N. Y. Filed Oct. 5, 1948. 4 claims. (Cl. 209–166.) Tall oil and derivatives are used.

2,496,052. Seam for woven wire fabrics. Alfred G. Hose and Donald C. Dilley, assignors to The Lindsay Wire Weaving Company, Cleveland, Ohio. Filed March 11, 1948. 4 claims. (Cl. 245–10.) The warp wires of bronze are sealed with a gold alloy.

2,496,122. Grained laminated structure. William T. Donahue, Chicago, Ill. Filed March 8, 1946. 1 claim. (Cl. 154–45.9.) A laminated base has a creped paper facing sheet and a transparent covering sheet.

February 7, 1950

2,496,336. Carton and method of sealing the flaps thereof. William J. Cupo, assignor to Waldorf Paper Products Co., St. Paul, Minn. Filed Sept. 1, 1944. 2 claims. (Cl. 229–37.) A method of sealing open ends of cartons.

2,496,349. Liner for adherent rubber surfaces and the like. Waldo Kellgren and Jesse J. Charpentier, assignors to Minnesota Mining & Manufacturing Company, St. Paul, Minn. Filed Sept. 26, 1945. 6 claims. (Cl. 206–59.) A creped paper liner for a pressure-sensitive adhesive tape is described.

2,496,440. Multiweb bonded paper products. George V. Caesar, Jordan V. Bauer, and Don M. Hawley, assignors to Stein, Hall & Company, New York, N. Y. Filed Nov. 14, 1946. 5 claims. (Cl. 154–141.) A quick-setting, waterproof adhesive for high-speed board machines contains 15–85% of starch gelatinized *in situ*, 5–50% of urea-formaldehyde resin, and 5–75% of hydrosol-forming materials.

2,496,471. High-pressure fluid sealing assembly. Lloyd Hornbostel, assignor to Beloit Iron Works, Beloit, Wis. Filed March 14, 1947. 14 claims. (Cl. 285–10.) A fluid-tight seal is applicable as a steam journal for a rotatable drier drum.

2,496,550. Methods of recovering chemicals in the manufacture of cellulose by digestion according to the alkaline sulphite and/or alkaline bisulphite methods. Eric J. Larsson and John H. Jonsson, assignors to Stora Kopperbergs Bergslags Aktiebolag, Falun, Sweden. Filed July 6, 1948. 6 claims. (Cl. 23–64.) The waste liquor is evaporated and burned and the aqueous solution is treated with carbon dioxide; the hydrogen sulphide is driven off by heating and sodium bicarbonate is crystallized from the solution.

2,496,566. Water-vapor resistant coated paper. Alexander Szwarc, Montreal, Canada. Filed Oct. 19, 1946. 2 claims. (Cl. 117-138.5.) A kraft paper is coated with a plasticized resin-asphalt wax compound.

2,496,731. Fresh produce package cooled by ice. Anthony Longo, Cranston, R. I., assignor to M. Longo & Sons Inc. Filed June 8, 1946. 3 claims. (Cl. 99-193.) A cardboard carton for fresh vegetables contains a waterproof bag containing ice.

2,496,753. Method of making rectangular flat bags. Leroy L. Salfisberg, assignor to Ivers-Lee Company, Newark, N. J. Filed Feb. 6, 1946. 1 claim. (Cl. 93-35.)

2,496,755. Frozen egg method and container. Louis Schwartzberg, Chicago, Ill. Filed April 9, 1945. 3 claims. (Cl. 99-196.) A carton for freezing the separated whites and yolks of eggs is coated on the interior with a wax-like nonadhering material.

2,496,796. Bag and method of making the same. Emanual S. Kardon, Melrose Park, Pa. Filed Oct. 11, 1947. 2 claims. (Cl. 229-59.) This covers a self-opening, satchel bottom-type paper bag.

2,496,856. Carbon paper. Charles B. Cook, Jr., West Hartford, Conn. Filed June 12, 1946. 2 claims. (Cl. 282–28.) A carbon paper is provided with 25 to 40 fine parallel corrugations per inch; they serve to prevent curling of the paper.

2,496,911. Method and apparatus for forming plastic laminates. Lawrence B. Green, assignor to Shellmar Products Corporation, Chicago, Ill. Filed June 7, 1945. 34 claims. (Cl. 154–37.) Paper and the like are impregnated with a polymerizable resin and the product is cured under a bank of heat lamps.

February 14, 1950

2,497,071. Package. William H. Colgate, Highland Park, Ill. Filed Nov. 21, 1944. 3 claims. (Cl. 206–45.31.) A container for the display of match boxes is described.

2,497,073. Reaction product of dicyandiamide with an amine-formaldehyde reaction product. James R. Dudley and John A. Anthes, assignors to American Cyanamid Company, New York, N. Y. Filed March 5, 1947. 2 claims. (Cl. 260–70.) The reaction product is useful in the preparation of wet-strength paper.

2,497,074. Modified urea-formaldehyde resins and methods of preparing the same. James R. Dudley and John A. Anthes, assignors to American Cyanamid Company, New York, N. Y. Filed March 5, 1947. 17 claims. (Cl. 260–29.4.) The products are useful as coating agents for the preparation of wet-strength paper.

2,497,124. Mechanism for handling production from cup forming machines or the like. Leo M. Harvey, La Canda, Calif. Filed March 28, 1944. 13 claims. (Cl. 93–60.)

2,497,203. Butter package. Clarence G. Bennett, San Mateo Calif., assignor to Paterson Pacific Parchment Company. Filed Nov. 12, 1947. 2 claims. (Cl. 99–179.) The use of vegetable parchment for packaging butter is claimed.

2,497,239. Cup-making machine. Frederick Ruza, assignor to Paper Container Manufacturing Co., Chicago, Ill. Filed March 22, 1944. 3 claims. (Cl. 93–36.2.)

2,497,318. Partition strip assembling machine. John E. McLaughlin, Julius E. Wilske, and Francis A. McCormick, assignors to Owens-Illinois Glass Company, Toledo, Ohio. Filed June 7, 1944. 21 claims. (Cl. 93–37.) A control of the assembling motor of a cardboard partition strip-assembling machine is described.

2,497,398. Oil base drilling fluid. Reginald D. Dawson, assignor to Shell Development Company, San Francisco, Calif. Filed May 24, 1947. 10 claims. (Cl. 252–8.5.) An emulsion is stabilized with magnesium tallate.

2,497,712. Process of manufacturing a fibrous sheet covered plywood. Richard J. Auchter, assignor to Paper Patents Company, Neenah, Wis. Filed June 5, 1944. 2 claims. (Cl. 154-132.) A resin-impregnated sheet is used for the plywood surface.

2,497,838. Starch treatment. John M. Newton and Lee D. Ough, assignors to Clinton Industries, Inc., Clinton, Iowa. Filed Nov. 16, 1948. 10 claims. (Cl. 195-99.) Starch is treated with phytic acid or its salts prior to enzymatic conversion for paper sizing.

February 21, 1950

2,497,882. Refined rosin products and processes. Burt L. Hampton, assignor to The Glidden Company, Cleveland, Ohio. Filed Feb. 23, 1946. 9 claims. (Cl. 260–105.) To prevent yellowing of paper sizes, rosin is heated at 200 to 375°C. with 0.5 to 5% of sulphur or a sulphur-containing compound for a time sufficient to decrease the degree of unsaturation, without aromatization, of substantially more than one ring of the rosin material.

2,498,005. Treatment of pigment comprising calcium carbonate. Harold R. Rafton, assignor to Raffold Process Corporation, Andover, Mass. Filed Oct. 31, 1946. 14 claims. (Cl. 23-193.) The properties of synthetic calcium carbonate are changed by grinding in a hammer mill.

2,498,197. Infusion package and method of making same. Thomas R. Baxter, assignor to Standard Brands Incorporated, New York, N. Y. Filed Jan. 9, 1948. 10 claims. (Cl. 99-77.1.) An infusion package of filter paper is overprinted with a network of lacquer.

2,498,207. Paper coating composition and coated paper. Herman C. N. Heckel, assignor to The Champion Paper & Fibre Company, Hamilton, Ohio. Filed Sept. 6, 1945. 2 claims. (Cl. 106–203.) The adhesive for the mineral pigment is an oxycellulose prepared with nitrogen dioxide.

2,498,213. Doctor device for rolls and cylinders. Ernst R. Ljungquist, assignor to Lodding Engineering Corporation, Worcester, Mass. Filed June 17, 1946. 2 claims. (Cl. 92-74.) Means are claimed for preventing a removable doctor blade from sliding out of its holder.

2,498,214. Doctor mechanism for paper machines. Ernst R. Ljungquist, assignor to Lodding Engineering Corporation, Worcester, Mass. Filed June 20, 1946. 1 claim. (Cl. 92-74.) Means are provided for moving a cylinder-roll doctor blade back and forth in a direction parallel to the axis of the roll.

2,498,224. Paper winding machine. William E. Stone, assignor to Rice Barton Corporation, Worcester, Mass. Filed Aug. 3, 1946. 4 claims. (Cl. 242-65.) A Pope reel paper-winding machine incorporates means for maintaining a constant nip pressure between a driving drum and a winding roll.

2,498,338. Pressure sensitive adhesive tape. John B. Martin, assignor to Kendall Company, Boston, Mass. Filed March 30, 1943. 6 claims. (Cl. 117-122.) The adhesive consists of rubber, reclaimed rubber, or a synthetic rubber, a tackifying resin, and a liquid plasticizer.

2,498,493. Electrically conducting composite sheet. Latimer F. Hickernell, assignor to Anaconda Wire and Cable Company, New York, N. Y. Filed June 22, 1945. 1 claim. (Cl. 174-126.) Paper impregnated with carbon black is laminated to a layer of metal foil with a special adhesive.

2,498,494. Electrical cable. Latimer F. Hickernell, assignor to Anaconda Wire and Cable Company, New York, N. Y. Filed June 22, 1945. 2 claims. (Cl. 174-26.) Each conductor is covered with a helical wrapping of paper impregnated with carbon black, with several wrappings of ordinary insulating paper, and then with a paper claimed in the above patent.

February 28, 1950

2,498,662. Calender roll. Le Roy Eaby, assignor to Armstrong Cork Company, Lancaster, Pa. Filed Dec. 31, 1946. 6 claims. (Cl. 257-95.) A calender roll is provided with a thin outer wall which results in a minimum temperature differential between the inside and outside surfaces.

2,498,757. Package container and display stand. Fred R. High, assignor to T. J. Holmes Co., Inc., Chartley, Mass. Filed Oct. 7, 1949. 8 claims. (Cl. 206-45.19.) A display package for glass atomizers is described.

2,498,786. Portable log-debarker having swingable frame mounting a driven feed wheel. Orezul Bouchard, Kirkland Lake, Ontario, Canada. Filed Sept. 30, 1946. 2 claims. (Cl. 144-208.)

2,498,844. Impact and acceleration testing. George R.

Sears, Robert D. Rae, and Johannes A. Van den Akker, assignors to The Institute of Paper Chemistry, Appleton, Wis. Filed Aug. 16, 1947. 4 claims. (Cl. 73-12.) A method and apparatus are described for calibrating and testing acceleration meters, impact recorders, "G" meters, and the like; it may be used also for testing containers and packing materials.

2,498,876. Stock controller. Robert E. Bowen, assignor to North Carolina Pulp Company, Plymouth, N. C. Filed Jan. 26, 1946. 12 claims. (Cl. 137-78.) A method is described for maintaining a uniform flow of stock at a uniform consistency.

2,499,187. Polymers of 2-vinyldibenzothiophene. Ralph G. Flowers and Leola W. Flowers, assignor to General Electric Company, Schenectady, N. Y. Filed June 17, 1948. 12 claims. (Cl. 260-79.7.) The polymers are useful for dielectrics, especially in paper-insulated capacitators.

2,499,267. Machine for making corrugated paperboard. William S. Conner, Atlanta, Ga. Filed Sept. 11, 1945. 5 claims. (Cl. 154-30.) Air or steam jets are used to strip the corrugated paper from one roll and to cause it to press against and follow a second roll.

2,499,313. Shaker dispenser. Roderick W. Hoag, Melrose, Mass. Filed June 22, 1945. 3 claims. (Cl. 229-7.) A corrugated paper package is used to dispense granular or powdered material.

March 7, 1950

R23,204. Carton. Julius A. Zinn, Jr., Chicago, Ill. Original No. 2,464,065, dated March 8, 1949. Application for reissue Nov. 3, 1949. 15 claims. (Cl. 229-37.)

2,499,416. Container with pouring throat and connected dispensing opening. George C. Reid and Samuel S. Jacobs, assignors to American Can Company, New York, N. Y. Filed May 22, 1947. 5 claims. (Cl. 229-17.)

2,499,430. Obtaining sterols of high purity. Henry A. Vogel and Roger M. Christenson, assignors to Pittsburgh Plate Glass Company, Allegheny County, Pa. Filed July 30, 1947. 24 claims. (Cl. 260-397.2.) The unsaponifiable fraction of tall oil (containing 20 to 50% sterol) in naphtha is extracted countercurrently with furfural.

2,499,461. Box sealing machine. Christian Christiansen, Swampscott, Mass. Filed April 23, 1943. 11 claims. (Cl. 93-36.3.) A method of sealing the glued ends of cardboard shoe boxes.

2,499,463. Paper strap. Jay D. Crary, assignor to Paper Strap, Inc., Portland, Oreg. Filed Feb. 12, 1946. 1 claim. (Cl. 154-53.6.)

2,499,528. Receptacle. Herbert L. Reitzes, Philadelphia, Pa. Filed Oct. 10, 1946. 7 claims. (Cl. 229-85.) A method of closing the flattened tubular end of a package.

2,499,564. Detector for carton closing machines. Joseph W. Binsack, assignor to Beech-Nut Packing Company, Canajoharie, N. Y. Filed Dec. 13, 1945. 6 claims. (Cl. 192-125.) The device is designed to stop the sealing machine or the feeding of the cartons in the event that a carton has not been properly sealed.

2,499,647. Box end closure. Louise James, assignor to Huye Space Saving Box System Inc., New Orleans, La. Filed Oct. 7, 1946. 1 claim. (Cl. 229-45.) A cardboard end closure incorporates locking means in a flange corner construction.

2,499,694. Conveying and positioning mechanism for cartons and the like. Richard C. Stenger, assignor to Sutherland Paper Company, Kalamazoo, Mich. Filed Jan. 10, 1949. 8 claims. (Cl. 198-31.) A conveyor mechanism for positioning and delivering cartons is adapted to deliver the cartons in two spaced rows.

2,499,780. Carton. George Rottman, Brooklyn, N. Y. Filed Sept. 5, 1945. 1 claim. (Cl. 229-31.) A cardboard hatbox.

2,499,799. Receptacle. William Traska, assignor to Electrolux Corporation, Old Greenwich, Conn. Filed Aug. 1, 1945. 2 claims. (Cl. 229-62.) A paper dust bag for a vacuum cleaner.

2,499,960. Carton assembling machine. John A. T. Leask, Detroit, Mich. Filed Aug. 27, 1945. 2 claims. (Cl. 93-36.3.) A machine is adapted to hold a hand-assembled carton during the glueing and drying operations.

2,500,041. Apparatus for changing coating solutions of coating machines. Leon P. Morey, assignor to Eastman Kodak Company, Rochester, N. Y. Filed Sept. 28, 1945. 16 claims. (Cl. 91-46.) A method is described for changing pans containing coating solutions.

March 14, 1950

2,500,408. Strip feed mechanism for paper cup making machines or the like. Leo M. Harvey, La Canada, Calif. Filed March 28, 1944. 8 claims. (Cl. 93-60.)

2,500,426. Moistureproofing compositions. Joe E. Moose, assignor to Monsanto Chemical Company, St. Louis, Mo. Filed May 24, 1946. 2 claims. (Cl. 106-270.) A moistureproofing composition for paper and the like consists of paraffin, ceresin, and *m*-terphenyl.

2,500,427. Moistureproofing compositions. Joe E. Moose, assignor to Monsanto Chemical Company, St. Louis, Mo. Filed Aug. 22, 1949. 2 claims. (Cl. 106-230.) A moistureproofing composition contains microcrystalline wax, *N*-*o*-xenylstearamide, and some hydrogenated rosin.

2,500,569. Machine for applying a liquid coating. Harry C. Rathke, assignor to The Meyercord Company, Chicago, Ill. Filed Jan. 3, 1947. 1 claim. (Cl. 91-51.) A method of applying a liquid adhesive to a decalcomania.

2,500,617. Electrosensitive stencil blank. Return J. Meigs, assignor to The Western Union Telegraph Company, New York, N. Y. Filed Aug. 4, 1942. 1 claim. (Cl. 117-35.5.) The stencil is impregnated with a waxy material mixed with carbon particles or a metal powder.

2,500,727. Treatment and purification of clay. Harry Whittaker, assignor to Georgia Kaolin Company, Elizabeth, N. J. Filed Aug. 6, 1949. 7 claims. (Cl. 23-112.) The clay is treated with an anion and then with a cation exchanger.

2,500,804. Carton feeding mechanism. Hugh F. Conser, assignor to Shedd-Bartush Foods, Inc., Detroit, Mich. Filed Jan. 5, 1946. 2 claims. (Cl. 93-53.) A mechanism for feeding flat articles (box blanks) from a stack and also for opening or squaring the blanks.

2,500,874. Package crimping machine. Leroy L. Salfisberg, assignor to Seal-Craft, Incorporated, Newark, N. J. Filed April 23, 1945. 6 claims. (Cl. 154-42.) A machine is adapted for crimping and sealing packages of small tablets (aspirin and the like).

2,500,848. Carrying device. Harold McMillan, assignor to Cohoes Envelope Company, Inc., Cohoes, N. Y. Filed Sept. 18, 1946. 2 claims. (Cl. 229-54.) A bellows-type paper bag is provided with an integral, reinforced handle.

March 21, 1950

2,500,922. Container flap folding apparatus. Samuel H. Berch, Beverly Hills, Calif. Filed Nov. 28, 1945. 3 claims. (Cl. 93-36.8.) A machine for packaging ice cream.

2,500,923. Apparatus for manufacturing cement-asbestos board. William Bernard and Joseph Bernard, assignors to American Asbestos Industries, Inc., South Orange, N. J. Filed June 6, 1946. 2 claims. (Cl. 92-43.)

2,500,972. Production of clay-coated sheet material. Alfred D. Wilson, assignor to S. D. Warren Company, Boston, Mass. Filed Feb. 14, 1945. 5 claims. (Cl. 106-148.) A method of forming a coating color from clay and casein.

2,501,180. Corrugated structure. Bernard P. Kunz, Denver, Colo. Filed April 1, 1946. 3 claims. (Cl. 154-55.) A series of corrugated sheets are glued together to form a structure which will withstand large compressive loads.

2,501,357. Pocket-size tissue container. Samuel S. Speckman and Murray Yoss, Bronx, N. Y. Filed Jan. 24, 1947. 2 claims. (Cl. 206-57.)

2,501,506. Pallet construction. Walter C. George, assignor to Gaylord Container Corporation, St. Louis, Mo. Filed June 13, 1945. 6 claims. (Cl. 248-120.) The construction of a fiberboard pallet for a lift truck is described.

2,501,540. Insulated drip baffle. Patrick P. Ryan, assignor to St. Regis Paper Company, New York, N. Y. Filed June 3, 1947. 5 claims. (Cl. 312-171.) A paper-base plastic is used as a thermal insulating member for refrigerators.

2,501,609. Container for flat articles. Stephen P. Midouhas, assignor to Kemline Metal Products Company, Bristol, Pa. Filed May 6, 1948. 5 claims. (Cl. 206-60.) A cardboard container has corner-reinforcing brackets.

March 28, 1950

2,501,665. Lignin resin and process of making same. Raymond N. Evans and Angelo P. Ingrassia, assignors to Masonite Corporation, Laurel, Miss. Filed May 24, 1946. 6 claims. (Cl. 260-17.3.) An acid-hydrolyzed lignocellulose fiber mat (63 parts) is impregnated with 113 parts of a resin solution containing 212 parts of Methyl Cellosolve and 150 parts of a melamine-formaldehyde condensation product; the product is heated 4.5 hours in a vacuum oven at 60°C., ground, and molded at 165°C. and 1750 p.s.i.

2,501,666. Lignin resins and process of making same. Raymond N. Evans, assignor to Masonite Corporation, Laurel, Miss. Filed Aug. 29, 1946. 4 claims. (Cl. 260-9.) An acid-hydrolyzed lignocellulose fiber stock at 3% consistency is treated with formaldehyde and a furfural-acetone condensation product.

2,501,694. Novelty box. Andrew E. Ruffin, Philadelphia, Pa. Filed March 28, 1947. 2 claims. (Cl. 229-8.) The walls are constructed of picture post cards.

2,501,716. Apparatus for manufacture of stereotype dry mats and similar multilayer sheet materials. Harold C. Friel, Hoosick Falls, N. Y. Filed July 5, 1946. 3 claims. (Cl. 92-43.)

2,501,811. Packaging for handled tools. John W. Cochran, assignor to Warwood Tool Company, Wheeling, W. Va. Filed

Oct. 28, 1949. 8 claims. (Cl. 206–65.) An L- or T-shaped corrugated board shipping container for handled tools.

2,501,842. Container. Robert B. Cartwright, Peoria, Ill. Filed Jan. 12, 1948. 14 claims. (Cl. 229-17.) A paper container for cereals and the like is provided with a pouring spout.

2,501,848. Log barker having water-jets oscillatable about longitudinal log axis. George M. Johnson, assignor to Rayonier Incorporated, New York, N. Y. Filed April 10, 1946. 4 claims. (Cl. 144-208.) High-velocity water jets cut through and remove the bark from the log without removing any of the wood.

2,501,849. Single closure for bottles. Charles H. Krebs, assignor to Standard Cap and Seal Corporation, Chicago, Ill. Filed May 22, 1947. 7 claims. (Cl. 215-38.) The closure is a laminate of paper, metal foil, and machine-glazed paper.

2,501,852. Means for effecting hermetic closures in cartons and containers. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed May 8, 1944. 6 claims. (Cl. 229-37.) A seam construction for heat-sealed cartons.

2,501,874. Photographic diazo-sensitized glassine paper. Willard D. Peterson, assignor to General Aniline & Film Corporation, New York, N. Y. Filed June 12, 1946. 2 claims. (Cl. 95-8.) A glassine paper is utilized as the base for a photographic diazotype material.

2,501,916. Arrangement for sorting paper pulp and similar suspended matters. Väinö V. Rantaniemi, assignor to Tammerfors Linne-och Jernmanufaktur Aktiebolag, Tammerfors, Finland. Filed Feb. 2, 1945. 3 claims. (Cl. 92-35.) A strainer for paper pulp and the like.

2,501,938. Machine for manufacturing spirally wound straws or tubes. Albert A. Heyman, assignor to Joseph Shapiro, Baltimore, Md. Filed Jan. 24, 1947. 6 claims. (Cl. 93-80.)

2,501,985. Web roll support for corrugated paperboard machines. Fred H. Benjamin, assignor to F. J. Kress Box Company, Pittsburgh, Pa. Filed July 2, 1947. 11 claims. (Cl. 242-58.) The roll can be loaded in the aisle beside the machine and moved into position.

2,502,080. Petroleum resin dispersion and the use thereof. Earl S. Fenelon, Jr., and Edward J. Pavilonis, assignor to Hercules Powder Company, Wilmington, Del. Filed April 19, 1947. 6 claims. (Cl. 106-218.) A molten mixture of wood rosin and petroleum resin is heated with 18% aqueous sodium carbonate; the dispersion is suitable as a paper sizing, an adhesive, etc.

2,502,111. Method of forming plural ply indented paper pads. Charles R. Walker, assignor to The Fox Paper Company, Cincinnati, Ohio. Filed Dec. 5, 1945. 7 claims. (Cl. 154-33.05.) An indented pad is formed of three webs of indented paper.

2,502,112. Indented paper manufacture. Charles R. Walker, assignor to The Fox Paper Company, Cincinnati, Ohio. Filed Dec. 5, 1945. 7 claims. (Cl. 154-33.05.) A multiple thickness padding of indented paper is formed by passage of a set of webs through a single pair of indenting rolls.

2,502,113. Indented paper pad. Charles R. Walker, assignor to The Fox Paper Company, Cincinnati, Ohio. Filed March 5, 1947. 4 claims. (Cl. 154-54.) A cushioning paper pad comprises at least three layers of identically indented paper webs.

2,502,117. Method and apparatus for lining blanks. Helmer Anderson, assignor to General Mills, Inc., Minneapolis, Minn. Filed March 17, 1948. 20 claims. (Cl. 93-36.01.) A continuous strip of lining material is fed to a series of spaced blanks, the lining material is cut between the blanks and then slitted to correspond to the slots in the blanks.

2,502,214. Container for carbon paper. Frank Gianninoto, assignor to Underwood Corporation, New York, N. Y. Filed Oct. 19, 1946. 7 claims. (Cl. 206-1.)

2,502,286. Laminated products and process for producing the same. Frank J. Sowa, Cranford, N. J. Filed July 25, 1945. 2 claims. (Cl. 154-140.) Asbestos sheets are laminated with an adhesive containing silicon compounds.

2,502,330. Washing paper pulp to remove coloring matter. Clarence E. Libby, Syracuse, N. Y. Filed March 1, 1943. 8 claims. (Cl. 92-13.) Pulp is washed with a dehydrated alkali metal phosphate.

2,502,340. Polyamide cold bonded laminate. John D. Pickens, assignor to E. I. du Pont de Nemours & Company, Wilmington, Del. Filed April 6, 1944. 6 claims. (Cl. 154–133.) Paper and the like is coated or impregnated with a solution of a *N*-alkoxymethyl polyamide.

2,502,384. Bottom construction for paperboard containers. Gaylord Loth, assignor to The Hinde & Dauch Paper Company, Sandusky, Ohio. Filed Sept. 4, 1948. 4 claims. (Cl. 229-39.)

UNITED STATES PATENTS ON PAPERMAKING

Second Quarter, 1950 — ***Compiled by CLARENCE J. WEST***

April 4, 1950

2,502,410. Method of bleaching cellulosic materials. Karl N. Cederquist, assignor to Mathieson Chemical Corporation, St. Louis, Mo. Filed April 22, 1946. 4 claims. (Cl. 8-104.) Pulp in an alkaline liquor is treated with chlorine dioxide, a part of which is transformed into a water-soluble chlorite, and the chlorite is activated to complete the bleaching action.

2,502,481. Duplex bag body. Amund S. Roper, assignor to Bemis Bro. Bag Company, Minneapolis, Minn. Filed Dec. 7, 1946. 1 claim. (Cl. 229-55.) The bag consists of an inner paper ply and an outer cellophane ply; the inner ply has a window opening.

2,502,498. Lignocellulose, phenol formaldehyde, and inorganic filler molding composition. Robert V. Williamson and Thomas F. Clark, assignors to United States of America as represented by the Secretary of Agriculture. Filed Oct. 18, 1946. 7 claims. (Cl. 260-17.2.) A molding composition contains about 50% lignocellulose, 25% phenol-formaldehyde resin, and 22.5-25% precipitated chalk.

2,502,561. Package device for shipping and displaying articles and display mantle therefor. Ludwig Ebert, assignor to Einson-Freeman Co., Inc., Long Island City, N. Y. Filed Feb. 25, 1947. 3 claims. (Cl. 206-44.) A corrugated shipping container for bottled goods.

2,502,586. Method of packaging lumped commodities. August F. Ottinger, assignor to Bemis Bro. Bag Company, St. Louis, Mo. Filed June 15, 1944. 1 claim. (Cl. 226-54.) The package consists of a cylindrical corrugated liner enclosed in an open-mesh bag.

2,502,732. Garment bag. Donald E. Lower, assignor of one half to Harry W. Lower, Butler Township, Pa. Filed May 27, 1946. 1 claim. (Cl. 206-7.)

2,502,763. Hydraulic-barker nozzle assembly producing composite oscillatory movement of discharging jet. Frank H. Swift, assignor to Crown Zellerbach Corporation, San Francisco, Calif. Filed Oct. 3, 1946. 5 claims. (Cl. 144-208.) Means are provided for barking logs with a minimum number of hydraulic barking nozzles without rotation of the log; the assembly can be used also for cants and slabs.

2,502,782. Method of coating paper. Donald R. Erickson, Kalamazoo, Mich. Filed Nov. 17, 1949. 2 claims. (Cl. 117-157.) An alkaline solution of hydroxyethylcellulose or hydroxypropylcellulose is used.

2,502,783. Coated paper and method of making same. Donald R. Erickson, Kalamazoo, Mich. Filed Nov. 17, 1949. 9 claims. (Cl. 117-157.) The coating color consists of Georgia clay dispersed with an alkaline solution of hydroxyethylcellulose.

2,502,883. Color stabilized rosin size. Edward N. Poor and Chester G. Landes, assignors to American Cyanamid Company, New York, N. Y. Filed May 12, 1948. 9 claims. (Cl. 106-218.) A mixture of 85-99% of pale rosin and 1-15% of wood rosin color bodies (previously heated in an oxygen-free atmosphere at 250 to 325° for 1 to 3 hours) is saponified with 9 to 16% of sodium carbonate.

2,502,884. Color stabilized rosin size. Edward N. Poor and Chester G. Landes, assignors to American Cyanamid Company, New York, N. Y. Filed May 12, 1948. 5 claims. (Cl. 106-218.) A mixture of 85-99% of pale rosin and 1-15% of tall oil pitch (previously heated in an oxygen-free atmosphere at about 300° for 2 hours) is saponified with sodium carbonate.

2,503,104. Process for precipitating silver from solutions. Eduard Farber, assignor to Timber Engineering Company, Washington, D. C. Filed May 13, 1946. 6 claims. (Cl. 75-108.) Silver is precipitated from dilute solution of complex salts by acid-hydrolyzed wood; the pH is adjusted to about 9.

2,503,210. Water transportation of wood. James O'Halloran, assignor to Anglo-Canadian Pulp and Paper Mills Limited, Quebec, Canada. Filed Feb. 10, 1947. 1 claim. (Cl. 214-152.) A method is described for loading wood onto ships.

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R23,218. Catalytic bleaching with chlorites. Robert M. Levy and James E. Brennan, assignors to Ecusta Paper Corpora-

tion, New York, N. Y. Original No. 2,477,631, dated Aug. 2, 1949. Application for reissue Feb. 13, 1950. 7 claims. (Cl. 8-105.)

2,503,240. Portable platform. Norman L. Cahners, assignor to Materials Handling Laboratories, Inc., Boston, Mass. Filed March 11, 1946. 3 claims. (Cl. 248-120.) The platform of a double-faced corrugated pallet is supported by a number of spaced corrugated members, adhesively attached to the under side of the platform.

2,503,267. Cigarette paper. Ward D. Harrison and Robinson E. Matthews, assignors to Ecusta Paper Corporation, New York, N. Y. Filed Sept. 16, 1944. 2 claims. (Cl. 92-3.) A cigarette paper containing 20 to 30% calcium carbonate is treated with 0.025 to 0.1% of urea-formaldehyde resin, which produces 50 to 100% increase in the wet strength of the paper.

2,503,297. Lignin aliphatic acids and salts. John S. Pierce, assignor of one half to The Albemarle Paper Manufacturing Company, Richmond, Va. Filed Dec. 3, 1945. 9 claims. (Cl. 260-124.) Alkali lignin is reacted with chloroacetic acid and alkali; the resulting ligninacetic acid is useful as a water-soluble insecticide, fungicide, wood preservative, sizing agent, or organic intermediate.

2,503,343. Collapsible egg carton. Henry Levine, Philadelphia, Pa. Filed Aug. 18, 1945. 1 claim. (Cl. 229-29.) This is an extension of U. S. patent 2,005,345 (June 18, 1935).

2,503,379. Article holding and display container. Stanley H. Davis, assignor to S. H. Davis Paper Box Company, Toledo, Ohio. Filed July 2, 1949. 2 claims. (Cl. 229-20.) The container has a cover which telescopes over the body and which may be swung laterally on its hinge to expose the open end of the box.

2,503,407. Method of making laminated fiberboard. Henry J. Perry, assignor to The J. P. Lewis Co., Beaver Falls, N. Y. Filed April 19, 1945. 6 claims. (Cl. 154-132.) The beater furnish includes 45% steamed groundwood, 15% kraft pulp, and 40% extracted pinewood pitch; 1 to 3% rosin size and 10 to 20% of a plasticizer (based on the resin) are added and the pH is adjusted to 4 or 5 with alum.

2,503,436. Suction box. Carleton L. Clark, New York, N. Y. Filed Sept. 21, 1945. 2 claims. (Cl. 92-52.)

2,503,454. Roofing felt. Robert G. Quinn, assignor to Johns-Manville Corporation, New York, N. Y. Filed Dec. 9, 1944. 2 claims. (Cl. 92-3.) The furnish contains 35-60% mechanically fiberized, steam softened wood fibers, 30-40% fiberized waste paper, and 0-25% mechanically fiberized rag fibers.

2,503,466. Apparatus for manufacturing cement-asbestos board. William Bernard and Joseph Bernard, assignors to American Asbestos Industries, Inc., Berkeley Heights, N. J. Filed Oct. 21, 1947. 7 claims. (Cl. 92-43.)

2,503,622. Dextran base wrinkle drying compositions. Enrique L. Luaces, assignor to New Wrinkle, Inc., Wilmington, Del. Filed May 20, 1949. 4 claims. (Cl. 106-215.) A dextran ether (e.g., ethyl, butyl, or β-hydroxyethyl) is added to the wrinkling oil.

2,503,623. Dextran base wrinkle drying compositions. Enrique L. Luaces, assignor to New Wrinkle, Inc., Wilmington, Del. Filed May 20, 1949. 4 claims. (Cl. 106-215.) A mixture of dextran ethers (e.g., benzyl and butyl or β-hydroxyethyl) is added to the wrinkling oil.

2,503,634. Dextran base wrinkle drying compositions. Enrique L. Luaces, assignor to New Wrinkle, Inc., Wilmington, Del. Filed May 20, 1949. 2 claims. (Cl. 106-215.) Dextran acetate is added to the wrinkling oil.

2,503,629. Web carrier and method of making same. Darwin Miscall, assignor to The Orr Felt and Blanket Company, Piqua, Ohio. Filed June 23, 1948. 6 claims. (Cl. 28-72.3.) A papermaking felt is impregnated with an alkylated melamine-formaldehyde resin.

2,503,681. Transfer ribbon manufacture. Douglas A. Newman, assignor to Columbia Ribbon and Carbon Manufacturing Company, Inc., Glen Cove, N. Y. Filed April 5, 1946. 11 claims. (Cl. 282-19.) A paper-base typewriter ribbon.

2,503,681. Transfer ribbon manufacture. Douglas A. Newman, assignor to Columbia Ribbon and Carbon Manufacturing Company, Inc., Glen Cove, N. Y. Filed March 26, 1946. 12 claims. (Cl. 197-172.) A paper-base typewriter ribbon.

2,503,772. Tall oil esters and process of making them. John B. Rust, assignor of one half to Montclair Research Corporation and one half to Ellis-Foster Company. Filed Oct. 26, 1946. 7 claims. (Cl. 260-22.) Tall oil and a polyhydric alcohol (e.g., glycerol) are heated at 180 to 300°C. and the resulting product is reacted with monoallyl maleate, phthalate, or the like.

2,503,798. Covered container. Kenneth T. Buttery, assignor to Sutherland Paper Company, Kalamazoo, Mich. Filed April 16, 1947. 7 claims. (Cl. 229-36.) A covered container has a moisture-resistant liner adhesively attached to the rear wall.

2,503,874. Flexible corrugated sheet material and apparatus. Charles Q. Ives, Reading, Mass. Filed Feb. 27, 1946. 22 claims. (Cl. 154-30.) The corrugations or flutes are interrupted at spaced intervals, with the top and side walls of the flutes terminating in the direction of the crest line at cut edges.

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2,504,179. Carton for ink bottles. Raymond Chemlinski, Schenectady, N. Y. Filed June 3, 1948. 4 claims. (Cl. 120-73.)

2,504,473. Machine for fabricating a cushioning packaging strip. Martin Van Antwerpen, Wauwatosa, Wis. Filed March 24, 1947. 16 claims. (Cl. 154-32.)

2,504,698. Machine for making roll dispensing containers. Jasper Kievit, assignor to The Crystal Waxing Company, Middletown, Ohio. Filed Sept. 28, 1948. 7 claims. (Cl. 1-2.)

2,504,744. Glass fiber sheet material. Reavis C. Sproull and Philip L. Staats, assignors to General Electric Company, Schenectady, N. Y. Filed June 3, 1944. 1 claim. (Cl. 92-3.) A sheet material suitable for capacitor dielectric purposes contains about 75% uncoated glass fibers and 25% kraft pulp.

2,504,920. Aqueous wax emulsion. Alfred F. Buckman, Jr., and Margaret M. Rendall, assignors to S. C. Johnson & Son, Inc., Racine, Wis. Filed March 18, 1947. 4 claims. (Cl. 260-23.) The wax emulsion is suitable for treating asbestos cement building boards.

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2,504,954. Logging arch. George S. Allin, assignor to Isaacson Iron Works, Seattle, Wash. Filed Dec. 22, 1947. 2 claims. (Cl. 214-65.3.) A logging arch is supported at opposite ends by large wheels.

2,505,034. Carton and display basket. John G. Fettinger, assignor to Robert Gair Company, Inc., New York, N. Y. Filed March 26, 1947. 5 claims. (Cl. 229-31.) The sides of an expansible carton can be pulled apart at the top when the cover is removed.

2,505,050. Apparatus for making paper cord. Benjamin M. Kimball, assignor to The Cellucord Corporation. Filed Sept. 7, 1948. 6 claims. (Cl. 57-35.)

2,505,192. Bottle carrier. Samuel N. Lebold, assignor to Morris Paper Mills, Chicago, Ill. Filed May 20, 1948. 3 claims. (Cl. 224-45.)

2,505,304. Lignin compounds and method of making same. Jörgen R. Salvesen and Carlyle Harmon, assignors to Marathon Corporation, Rothschild, Wis. Filed June 19, 1947. 4 claims. (Cl. 260-124.) A demethylated lignin compound is prepared by heating sodium lignosulphonate and sodium hydroxide at 265 to 325°C. for 30 to 120 minutes.

2,505,399. Combined bottle shipping and display device. Grover C. Hilton, Hutchinson, Kans. Filed Feb. 24, 1948. 2 claims. (Cl. 206-45.14.) A device for liquid cement.

2,505,404. Machine for corrugating cardboard. Peter Jacobsen, assignor to Dry Corrugating Method v/akselbo, Berbom og Drostholm, Copenhagen, Denmark. Filed March 21, 1947. 12 claims. (Cl. 154-31.) Two toothed cooperating rollers are used, the diameters of which are such that not more than three teeth on one roller are engaged with the teeth of the other roller at any one time.

2,505,442. Carton. Manville Thomas, assignor to Marathon Corporation, Rothschild, Wis. Filed Oct. 22, 1947. 5 claims. (Cl. 229-33.) A collapsible container for frozen foods.

2,505,513. Method of and apparatus for screening paper pulp. Frank W. Young and Sven A. Fahlgren, assignor to Bird Machine Company, South Walpole, Mass. Filed Feb. 1, 1944. 3 claims. (Cl. 92-33.) The knotter for raw unwashed stock is so operated that air will not be mixed with the liquor flowing to and through it. The stock is introduced into the liquor below the liquid level in the knotter box.

2,505,545. Capacitor paper. Richard J. Hopkins and Reavis C. Sproull, assignors to General Electric Company, Schenectady, N. Y. Filed Oct. 16, 1944. 2 claims. (Cl. 175-41.) The base is a kraft paper containing 91-95% cellulose, 1-5% pentosans, 0.5–5% lignin, and 0.15–0.30% ash, which is impregnated with a liquid dielectric material.

2,505,697. Dispensing container. Edward E. Vomacka, Glendale, Mo. Filed April 15, 1946. 3 claims. (Cl. 222-361.)

2,505,703. Carton erecting machine. Leon Benoit and Raymond Vahle, assignors to Robert Gair Company, Inc., New York, N. Y. Filed Dec. 5, 1945. 11 claims. (Cl. 93-37.)

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2,505,748. Lid for paper containers. Cesare Barbieri, assignor to Dixie Cup Company, Chicago, Ill. Filed Aug. 12, 1948. 4 claims. (Cl. 229-5.5.) The lid is so constructed that it can be adjusted to fit receptacles of various sizes and shapes.

2,505,782. Sulphur containing molding compositions, sulphur containing consolidated products, and methods of making the same. John G. Meiler and Karl B. Bollerslev, assignors to Plywood Research Foundation, Tacoma, Wash. Filed June 15, 1948. 14 claims. (Cl. 260-17.2.) The molding composition

consists of finely divided lignocellulosic material, a thermosetting phenol-aldehyde resin, and sulphur.

2,505,818. Solubilizing tanning bark extracts with waste sulphite liquors. Kenneth T. Williams and Earl F. Potter, assignors to the United States of America as represented by the Secretary of Agriculture. Filed July 23, 1946. 5 claims. (Cl. 8-94.31.) The extract from western hemlock bark is heated with sodium lignosulphonate 3 hours at about 180°F.; the resulting product is a tanning agent.

2,505,886. Molded pulp cushion pad. William J. De Reamer, assignor to Mapes Consolidated Manufacturing Company, Griffith, Ind. Filed May 6, 1947. 3 claims. (Cl. 217-26.) A molded pulp cushion pad is designed for use with fillers in a standard egg case.

2,505,926. Poultry head. Emmitt W. West and Edith E. West, Holbrook, Ariz. Filed April 22, 1947. 3 claims. (Cl. 229-88.) The hood is made of a flexible oil paper.

2,506,021. Machine for wrapping folded sheets. John A. Holmberg, assignor to Crown Zellerbach Corporation, San Francisco, Calif. Filed Feb. 15, 1947. 4 claims. (Cl. 93-93.) The machine compresses and wraps bundles of folded tissue as they are delivered from the folding machine.

2,506,048. Creep testing apparatus. Johannes A. Van den Akker, assignor to The Institute of Paper Chemistry, Appleton, Wis. Filed Oct. 23, 1946. 4 claims. (Cl. 73-100.) The apparatus is designed for testing the permanent deformation or creep of paperboards resulting from prolonged stress.

2,506,056. Gastight and gas-filled package and method of making it. Samuel Bergstein, Cincinnati, Ohio. Filed Oct. 6, 1945. 8 claims. (Cl. 229–3.5.) A carton is so constructed that it is rendered gastight by dipping only the ends. Thus, the whole package is not subjected to the heat of the molten coating bath.

2,506,057. Means and method for rendering paperboard cartons gastight and packages so produced. Samuel Bergstein, Cincinnati, Ohio. Filed Oct. 25, 1945. 12 claims. (Cl. 229-3.5.) A glassine sleeve surrounds the carton and two caplike members enclose the ends of the package.

2,506,153. Opacification of paper. Albert B. Hurley, assignor to North American Paper Process Company, Inc., Dover, Del. Filed June 17, 1947. 4 claims. (Cl. 88-1.) A mixture of titanium dioxide, casein, and glycerin is used to coat sheets of relatively translucent paper with a checkerboard pattern on both sides of the sheets.

2,506,161. Die holder. Lester H. Messinger, assignor to Remington Arms Company, Inc., Bridgeport, Conn. Filed June 14, 1947. 3 claims. (Cl. 93-83.) A die holder is provided for use with paper tube-sizing machines.

2,506,311. Bag pouch. George A. Moore, assignor to Reynolds Metals Company, Richmond, Va. Filed Sept. 11, 1946. 4 claims. (Cl. 229-62.) A tobacco pouch is formed from a laminate of paper and aluminum foil.

2,506,346. Pallet construction. Madison L. Crawford, assignor to Clark Equipment Company, Buchanan, Mich. Filed Jan. 25, 1946. 1 claim. (Cl. 248-120.) The construction of a paperboard pallet is described.

2,506,478. Instrument for testing paper. Alan V. Wright, assignor to Kelvin, Bottomley & Baird, Limited, Glasgow, Scotland. Filed June 5, 1947. 4 claims. (Cl. 175-183.) This patent covers the K.B.B. sizing tester.

2,506,550. Sheet conveying, stacking, and delivering apparatus. Hugh S. Morrison, assignor to Fairfield Paper and Container Company, Baltimore, Ohio. Filed Dec. 27, 1948. 16 claims. (Cl. 214-6.) The apparatus is adapted to paperboard.

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2,506,667. Composite textile yarn for use in papermaking felts. Edward H. Hall, Fitchburg, Mass. Filed Nov. 24, 1948. 5 claims. (Cl. 57-140.) Nylon fiber is incorporated into an asbestos yarn.

2,506,678. Apparatus for use in making paper. Harry W. Osgood, Jr., assignor to S. D. Warren Company, Boston, Mass. Filed July 6, 1944. 5 claims. (Cl. 92-44.) This patent covers a slice.

2,506,820. Closure structure. Ernest C. Webb, Lexington, Ky. Filed Sept. 17, 1946. 9 claims. (Cl. 229-44.) A metal barb on the steel cover for cooky display containers pierces the wall of a cardboard carton from the inside and engages the cover to the wall.

2,507,080. Ball plant container. Ralph E. Acker and Paul A. Nemoede, assignors to Container Corporation of America, Chicago, Ill. Filed Sept. 16, 1948. 6 claims. (Cl. 229-27.) A container for plants, shrubs, and the like which have balls of earth attached.

2,507,144. Web winding machine. Peter J. Christman, Green Bay, Wis. Filed April 2, 1948. 13 claims. (Cl. 242-56.) The machine converts a web of sheet material into relatively small rolls (toilet paper, towels, and the like).

2,507,197. Diaper. Hilda T. S. de Matzdorf, Guatemala, Guatemala. Filed July 7, 1948. 3 claims. (Cl. 128-287.) The construction of a disposable diaper is described.

2,507,237. Process of treating crude sulphate soap skimmings. Clarence B. F. Young and Edward J. Calleton, assignors to National Southern Products Corporation, New York, N. Y. Filed Jan. 9, 1947. 5 claims. (Cl. 260-97.7.) The skimmings are spray dried by atomization into air at 400 to 700°F.; the dried material has a relatively inoffensive odor.

2,507,403. Carton. Fred C. Gluck, assignor to Minerva Wax Paper Company, Minerva, Ohio. Filed Aug. 13, 1945. 7 claims. (Cl. 206–58.) A hexagonal carton is adapted for dispensing wax paper from a roll.

2,507,404. Carton. Fred C. Gluck, assignor to Minerva Wax Paper Company, Minerva, Ohio. Filed Aug. 24, 1945. 4 claims. (Cl. 206-58.) A cover is claimed for a dispensing container for wax paper in roll.

2,507,418. Coating machine for sheets of materials. Harry W. Osgood, Jr. and Alexander Jones, assignors to S. D. Warren Company, Boston, Mass. Filed Sept. 30, 1946. 4 claims. (Cl. 91-33.) A machine for coating sheets for test purposes.

2,507,430. Carton and closure therefor. Charles R. Yancey, Dallas, Texas, assignor of one half to Katherine K. Yancey. Filed Sept. 11, 1944. 12 claims. (Cl. 229-17.) A container with a pouring spout.

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R23,230. Valve bag. Harry E. Lee, assignor to St. Regis Paper Company, New York, N. Y. Original No. 2,478,113, dated Aug. 2, 1949. Application for reissue Jan. 27, 1950. 3 claims. (Cl. 229-62.5.)

2,507,489. Egg carton filling apparatus. Kenneth T. Buttery and Eugene Hubbard, assignors to Sutherland Paper Company, Kalamazoo, Mich. Filed Oct. 15, 1945. 3 claims. (Cl. 226-14.)

2,507,588. Load supporting pallet. Martin Brandon, Franklin P. Robinson, and Larry J. Budd, assignors to Pallet Devices, Inc., Chicago, Ill. Filed Jan. 29, 1947. 7 claims. (Cl. 248-120.) A paperboard pallet has a hollow pillar construction which permits a supported load to be banded to the pallet.

2,507,626. Method of making coin wrappers. Ernest W Ekstrand, assignor to Ekstrand Mfg. Co., Inc., Orange, N. J. Filed Nov. 1, 1943. 1 claim. (Cl. 93-1.)

2,507,824. Box. John C. Simpson, Portland, Oreg., assignor of one half to L. B. Sandblast. Filed Feb. 10, 1947. 3 claims. (Cl. 229–45.) The lid is adapted to be opened and closed during inspection of the contents.

2,507,827. Filtering material. Earl Stafford and Horace N. Lee, assignors to Arthur D. Little, Inc., Cambridge, Mass. Filed Jan. 27, 1944. 13 claims. (Cl. 183-44.) A sheet material for filtration of gases and vapors consists of 5% long fibers (caroa) and 95% of equal parts of unhydrated cut cotton fibers and cut viscose fibers, which are combined with 4–8% asbestos fibers.

2,507,843. Convertible container. Leonard A. Wheeler, Los Angeles, Calif. Filed April 23, 1946. 2 claims. (Cl. 229-1.5.) A heavy paper container for popcorn can be converted to a megaphone.

2,507,929. Insulated package. Lewis C. Pennebaker, assignor to O. B. Andrews Company, Chattanooga, Tenn. Filed June 14, 1946. 6 claims. (Cl. 229-14.) A package consists of inner and outer paperboard containers spaced apart by fillers or supports; the space is filled with dry ice or other media.

2,507,978. Anchoring means for sheet material members. Joseph E. Jubin, New Orleans, La. Filed Dec. 2, 1946. 5 claims. (Cl. 229-45.) Locking tabs on one member are inserted through cooperating slots in a second member.

2,508,043. Process for making battery separators. John F. Schaefer, assignor to Globe-Union Inc., Milwaukee, Wis. Filed Dec. 28, 1944. 1 claim. (Cl. 92-21.) Mechanical pulp is heated with 1–5% sodium hydroxide at atmospheric pressure and the slurry at 160°F. is adjusted to pH 1–3 with sulphuric acid; the acid-free product is formed into a sheet and heated to 250–450°F.

2,508,045. Moisture indicator for webs. John S. Seney, assignor to E. I. du Pont de Nemours & Company, Wilmington, Del. Filed Oct. 8, 1946. 13 claims. (Cl. 175–183.) The indicator is responsive to changes in the electrical resistance of the web.

2,508,083. Stripper. Elmer L. Winkler, Collinsville, Ill Filed June 3, 1946. 2 claims. (Cl. 93-58.) The apparatus automatically strips the waste portion from box blanks which have been incompletely severed from cardboard sheets.

2,508,119. Method of operating heat exchangers. Carl J. Lockman, assignor to Rosenbald Corporation, New York, N. Y. Filed May 12, 1945. 3 claims. (Cl. 257-1.) A method of evaporating sulphite waste liquor.

2,508,193. Reclosable carton. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Feb. 23, 1946. 7 claims. (Cl. 229-17.) A seal-end carton is described.

2,508,287. Process for coating paper. Charles R. Outterson and Benjamin A. Ford, assignors to Albemarle Paper Manufacturing Company, Richmond, Va. Filed Oct. 14, 1946. 2 claims. (Cl. 117-64.) The coated paper web is passed over an inclined path while the coating composition is still fluid.

May 23, 1950

2,508,528. Foam separator. John A. McPherson, assignor to Mosinee Paper Mills Company, Mosinee, Wis. Filed Jan. 12, 1949. 6 claims. (Cl. 252-361.) The separator has an outlet for discharging the quiescent liquid, an outlet for the released gases, and a series of spaced, corrugated baffles for contacting the foaming liquid.

2,508,579. Garment packing device. William W. McFall, River Forest, Ill. Filed Feb. 14, 1948. 7 claims. (Cl. 206-7.) A paperboard garment container.

2,508,594. Hydraulic type bark-removing machine having nozzle adjustable about two spaced horizontal axes. Harry E. Bukowsky, assignor to Crown Zellerbach Corporation, San Francisco, Calif. Filed Dec. 3, 1945. 1 claim. (Cl. 144-208.) A barker suitable for logs of different diameters.

2,508,629. Method of feeding raw materials to smelting furnaces. Ragnar Tanberg, Drammen, Norway. Filed Sept. 13, 1946. 3 claims. (Cl. 49-77.) Raw material to be fed to glass-smelting furnaces is bonded with evaporated sulphite waste liquor.

2,508,754. Parachute container of folded paper. Leonard P. Frieder, Great Neck, N. Y., and Walter S. Finken, said Finken assignor to said Frieder. Filed Aug. 17, 1945. 13 claims. (Cl. 244-148.)

2,508,798. Machine for making collapsible and expansible tubes. Samuel D. Polsen and Sven H. Nelson, assignors of one fourth each to Blanche H. Polsen, Mildred P. Nelson, Sven H. Nelson, and Norman H. Nelson, Bridgeport, Conn. Filed Oct. 18, 1945. 15 claims. (Cl. 93-80.) A strip of paper is formed into an expansible and collapsible bellow-like tube.

2,508,909. Packaging receptacle with latching handle. Richard J. Evans and James B. Barrow, Berkeley, Calif.; said Barrows assignor to said Evans. Filed March 2, 1949. 18 claims. (Cl. 229-52.) A container for baked goods is provided with cover flaps.

2,508,943. Bottle carrier. Maynard G. Hall and Michael H. Kowal, assignors to Empire Box Corporation, Garfield, N. J. Filed May 6, 1948. (Cl. 229–28.)

2,508,962. Container. George A. Moore, New York, N. Y. Filed June 23, 1945. 2 claims. (Cl. 229-1.5.) A three-sided wedge-shaped container.

2,509,125. Glued bag and method of producing the same. Clyde K. Billeb, assignor to Milprint, Inc., Milwaukee, Wis. Filed June 21, 1947. 3 claims. (Cl. 229-57.) A triple bottom seal is designed for the bottom of the bag.

2,509,142. Testing machine for rupturing specimens. Raymond E. Getchell, assignor to B. F. Perkins & Son, Inc.. Holyoke, Mass. Filed Oct. 16, 1948. 2 claims. (Cl. 72-102.) A bursting strength tester is provided with means for clamping the specimen and subjecting it to the action of a hydraulically loaded rubber diaphragm.

2,509,143. Diaphragm construction for specimen bursting machines. Raymond E. Getchell, assignor to B. F. Perkins & Son, Inc., Holyoke, Mass. Filed Jan. 24, 1949. 2 claims. (Cl. 137-157.) The rubber diaphragm of a hydraulic-type bursting strength tester offers no resistance in itself to the action of fluid pressure, so that the pressure of the fluid at the moment of rupture of the specimen is the true result.

May 30, 1950

2,509,289. Carton pouring spout. Robert M. Dunning, assignor to Waldorf Paper Products Company, St. Paul, Minn. Filed Sept. 26, 1946. 2 claims. (Cl. 229-17.) A pouring spout is formed from three adjoining panels of a carton.

2,509,296. Cylinder machine. Philip H. Goldsmith, Wilmington, Del. Filed Feb. 3, 1945. 15 claims. (Cl. 92-43.) A mold construction is proposed by means of which a vacuum can be applied substantially throughout the length of the mold in advance of the point of pickup to prevent crushing at the couch nip.

2,509,447. Carton and carton blank. Charles J. Prawdzik, assignor to Union Bag and Paper Corporation, New York, N. Y. Filed July 17, 1947. 3 claims. (Cl. 229-37.) A corrugated shipping container is tapered at its vertical cross section.

2,509,450. Bacon package. Harold B. Reed, Fred W. Swanson, and Leo Peters, said Reed and Peters assignors to Swanson, Park Ridge, Ill. Filed May 8, 1948. 8 claims. (Cl. 99-174.)

2,509,468. Furniture corner protector. Wilbur G. Anderson, Jr., assignor to American Box Board Company, Grand Rapids, Mich. Filed July 11, 1949. 7 claims. (Cl. 229-14.) The material is slotted and scored so that it can be folded quickly into a three-sided protector.

2,509,499. Matrix sheet and process of preparing same. Sigfried Higgins, assignor to Union Carbide and Carbon Corporation, New York, N. Y. Filed June 26, 1947. 10 claims. (Cl. 18-47.5.) A thermosetting resin-fiber matrix sheet is brush coated on one side with hydroxyethylcellulose.

2,509,616. Carton. Bennie A. Rafoth and George E. Wauda, assignors to Marathon Corporation, Rothschild, Wis. Filed Nov. 14, 1947. 1 claim. (Cl. 229-36.) An ice cream carton is provided with a pocket for a wooden spoon.

2,509,822. Stock inlet. Lloyd Hornbostel, assignor to Beloit Iron Works, Beloit, Wis. Filed Dec. 21, 1946. 12 claims. (Cl. 92-44.) A method of maintaining a constant stock level in a head box.

2,509,884. Tall oil treatment. Ronald Rosher, assignor to Hercules Powder Company, Wilmington, Del. Filed May 24, 1947. 9 claims. (Cl. 260-97.6.) Tall oil is selectively esterified, the mixture is dissolved in a hydrocarbon solvent, and the resin acids, are precipitated by cyclohexylamine.

2,510,004. Telescopic shipping carton. Benjamin M. Williams, assignor to Gaylord Container Corporation, St. Louis, Mo. Filed Nov. 1, 1947. 1 claim. (Cl. 229-14.) A relatively tall heavy-duty carton.

2,510,034. Manufacture of chlorine dioxide. John F. Haller, assignor to Mathieson Chemical Corporation, New York, N. Y. Filed Jan. 13, 1948. 4 claims. (Cl. 23-152.) Chlorine dioxide is prepared from a water-soluble metal chlorate and a reducing agent at a pH below 2 and with a 10% chlorate concentration.

June 6, 1950

2,510,120. Masking paper. Russell J. Leander, Chicago, Ill. Filed May 31, 1946. 2 claims. (Cl. 117-44.) A masking paper is impregnated with rubber or a similar material to render it stretchable (up to 15%) in two dimensions.

2,510,177. Hot melt coating composition. William Horback, assignor to Celanese Corporation of America, New York, N. Y. Filed Nov. 29, 1946. 2 claims. (Cl. 106-181.) The composition consists of 100 parts of cellulose propionate, 90 parts dibutyl sebacate, and 10 parts butoxyethyl stearate.

2,510,211. Dual-purpose container. William H. Cleary, assignor of one half to John D. Shourt, Minneapolis, Minn. Filed Aug. 6, 1947. 3 claims. (Cl. 229-15.) The container is chemically treated to withstand baking temperatures.

2,510,213. Geographical globe. John W. Ekstedt, Union, Albert F. Pityo, Clifton, and Harry Butterfield, Maplewood, N. J. Filed March 28, 1945. 25 claims. (Cl. 35-46.) The globe is formed of paper coated or impregnated with a thermosetting resin.

2,510,214. Method of and apparatus for forming blanks. John W. Ekstedt, Union, Albert F. Pityo, Clifton, and Harry Butterfield, Maplewood, N. J. Filed Feb. 17, 1947. 9 claims. (Cl. 18-19.) The paper blank for the globe consists of a smooth, hard outer layer and a highly porous, absorbent inner layer; they are impregnated with a thermosetting phenolic resin.

2,510,215. Method of forming hemispherical globe sections. Albert F. Pityo, Clifton, and Harry Butterfield, Maplewood, N. J. Filed May 12, 1947. 4 claims. (Cl. 154-110.) The sections consist of an inner layer of cardboard, an intermediate layer of paper, and an outer layer of a plastic material, such as cellulose acetate.

2,510,251. Machine for setting up box blanks. Louis Perilli, assignor to Associated Folding Box Co., South Boston, Mass. Filed Feb. 15, 1947. 19 claims. (Cl. 93-53.)

2,510,254. Suction filter. Johan C. F. C. Richter, assignor to Aktiebolaget Kamyr, Karlstad, Sweden. Filed Nov. 26, 1945. 5 claims. (Cl. 210-199.) A sieve drum is mounted in a tank so that it is partially immersed in the pulp mixture; the interior of the drum forms a closed suction chamber.

2,510,401. Machine for testing snap and recovery of sheet material. Charles E. Johnson, assignor to Brown & Bigelow, St. Paul, Minn. Filed Dec. 22, 1947. 6 claims. (Cl. 73-100.) A testing method for the resiliency and fatigue of paper.

2,510,530. Package spout. William F. Telschow, assignor to Colgate-Palmolive-Peet Company, Jersey City, N. J. Filed June 18, 1945. 8 claims. (Cl. 222-81.) A device for puncturing a pouring opening in the wall of a cardboard container.

2,510,595. Method of bleaching groundwood. Robert L. McEwen and Fred R. Sheldon, assignors to Buffalo Electro-Chemical Company, Inc., Tonawanda, N. Y. Filed Sept. 20, 1946. 2 claims. (Cl. 8-104.) A partially dewatered pulp sheet is treated on one face with an alkaline solution of hydrogen peroxide and an alkali metal phosphate.

2,510,661. Loading paper fibers with organopolysiloxane coated clay. Moyer M. Safford, assignor to General Electric Company, Schenectady, N. Y. Filed May 6, 1944. 3 claims. (Cl. 92-21.) Paper stock is treated in the beater with a clay which has been coated with an organopolysiloxane.

2,510,755. Purification of abietic acid. Arthur Pollak and

Henry P. Johnston, assignors to West Virginia Pulp and Paper Company, New York, N. Y. Filed Dec. 19, 1946. 3 claims. (Cl. 260-97.6.) The crude crystalline abietic acid is washed with 0.5% sodium carbonate at 120°F.

2,510,760. Carton protector. Louis Schott, Cincinnati, Ohio. Filed Jan. 18, 1946. 2 claims. (Cl. 229-23.) A metal protector is placed under stored, filled cartons.

2,510,776. Air-entrained concrete and method of making. Carl O. Gabrielson, assignor to Mo och Domsjo Aktiebolaget, Ornskoldsvik, Sweden. Filed Dec. 6, 1948. 2 claims. The plasticity of a concrete or mortar mix is enhanced by the addition of not more than 0.1% by weight (of the cement) of alkali-treated tall oil pitch.

June 13, 1950

2,510,941. Method of brittlizing pencil sheaths. John E. Ake and Kay Miller, assignors to Joseph Dixon Crucible Company, Jersey City, N. J. Filed April 22, 1949. 6 claims. (Cl. 154-117.) Pencils having a convolute sheath of paper secured by adhesives are heated at 390 to 400°F.

2,511,031. Method and apparatus for forming paper bags. Stanley G. Yount, Los Angeles, Calif. Filed Aug. 9, 1946. 3 claims. (Cl. 93-35.) Method of forming pleated tubes from two continuous rolls.

2,511,196. Production of cellulose. Stanley C. Bate, assignor to Celanese Corporation of America, New York, N. Y. Filed Oct. 11, 1944. 9 claims. (Cl. 92-9.) Wood is digested with a mixture of nitric and acetic acids for 6 hours at 70 to 110°C. while oxygen is passed through the mixture.

2,511,189. Drip tray. William W. Woodward, assignor to The Fairfield Paper and Container Company, Baltimore, Md. Filed Aug. 23, 1949. 5 claims. (Cl. 229-23.) The construction of a paperboard drip tray is claimed.

2,511,303. Window bag and method and apparatus for making same. Henry W. Stevens and Leonard R. Hecker, assignors to Benj. C. Betner Company, Devon, Pa. Filed Sept. 9, 1946. 3 claims. (Cl. 154-37.)

2,511,317. Handle for bottle carrying containers. Edwin L. Arneson, assignor to Morris Paper Mills, Chicago, Ill. Filed July 7, 1948. 1 claim. (Cl. 229-52.)

2,511,415. Web coating apparatus. Charles P. Putnam and Webster E. B. Baker, assignors to New York and Pennsylvania Co., Inc., New York, N. Y. Filed May 16, 1945. 8 claims. (Cl. 91-51.) Excess coating is removed from the paper web by air streams.

2,511,417. Machine for manufacturing paper plant protectors. Walter R. Schindler, assignor to Hot Kap Manufacturing Company, Los Angeles, Calif. Filed Dec. 31, 1948. 7 claims. (Cl. 93-1.)

2,511,442. Dispensing container. Jacob O. Lundberg, Stockholm, Sweden. Filed Dec. 20, 1946. 1 claim. (Cl. 206-57.) A container for a stack of paper sheets.

2,511,481. New-type drum package. Eugene S. Schneider, assignor to Rohm & Haas Company, Philadelphia, Pa. Filed May 11, 1949. 5 claims. (Cl. 229-14.) A container for liquids.

2,511,523. Self-locking carton. Albert A. Abrams, St. Louis, Mo. Filed March 18, 1947. 4 claims. (Cl. 229-33.)

2,511,542. Flatware tray. Eric J. Rau, assignor to Container Corporation of America, Chicago, Ill. Filed Aug. 14, 1948. 9 claims. (Cl. 206-45.19.) A traylike paperboard container and insert construction.

2,511,550. Ventilated shipping container. Harold A. Simms, assignor to The Patent and Licensing Corporation, New York, N. Y. Filed June 7, 1946. 1 claim. (Cl. 229-15.) A chick box of foldable sheet material.

2,511,569. Article holding insert for containers. Stanley H. Davis, Louis F. Middlesworth, and Arthur L. Milstein, assignors to The S. H. Davis Paper Box Company, Toledo, Ohio. Filed July 2, 1949. 15 claims. (Cl. 206-65.) A stiff paper liner is used in containers for hollow glassware or the like.

2,511,620. Structural panel. MacMillan Clements, Southport, Conn. Filed April 16, 1948. 2 claims. (Cl. 20-91.) A hollow door panel is made of corrugated board.

2,511,783. Planographic printing sheet. Douglas A. Newman, assignor to Columbia Ribbon and Carbon Manufacturing Company, Inc., Glen Cove, N. Y. Filed Feb. 28, 1946. 10 claims. (Cl. 101-149.2.) A laminated planographic printing sheet comprises two layers of paper which are parchmentized on only one side; the unparchmentized sides are bonded together by means of a waterproof adhesive.

2,511,816. Laminate. Elgin L. Shaw, assignor to Goodyear Aircraft Corporation, Akron, Ohio. Filed Jan. 19, 1946. 1 claim. (Cl. 154-45.9.) A solid vulcanized fiber core is used with metal sheets.

June 20, 1950

2,511,911. Process of preparing flameproofing glycol monosulphates. Walter M. Fuchs, New York, N. Y., and Eskil Gavatin; said Gavatin assignor to said Fuchs. Filed Jan. 5, 1949. 7 claims. (Cl. 260-458.) Directions are given for the preparation of glycol monosulphates with two to six carbon atoms, which are suitable flameproofing agents.

2,512,111. Cradle egg carton. Clement L. Monahan, Chicago, Ill. Filed Jan. 23, 1947. 2 claims. (Cl. 229-28.)

2,512,168. Paper roll. Robert C. Moore, Long Island City, N. Y. Filed March 8, 1949. 3 claims. (Cl. 206-58.) A roll for a paper-dispensing pencil is provided with scored weakening lines so that a clean tear of paper from the roll is assured.

2,512,311. High-frequency heating apparatus. Maurice F. Davis, assignor to General Electric Company, Schenectady, N. Y. Filed Sept. 1, 1948. 5 claims. (Cl. 219-47.) A method of drying paper and like materials in a high-frequency apparatus.

2,512,347. Dielectric stencil. Allen R. Lindsay, Cleveland Heights, Ohio. Filed Sept. 23, 1948. 4 claims. (Cl. 101-128.2.) The basis is a sheet of uncoated long fiber open tissue which has been treated with a chlorinated oil.

2,512,348. Method of making dielectric stencils. Allen R. Lindsay, Cleveland Heights, Ohio. Filed Sept. 23, 1948. 5 claims. (Cl. 101-128.2.) The basis is a tissue paper coated with film of Buna rubber.

2,512,382. Method of forming collapsible containers. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Sept. 8, 1945. 5 claims. (Cl. 93-51.) The containers are suitable for frozen foods, ice cream, and the like.

2,512,383. Container for solid and liquid food products. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Nov. 10, 1947. 8 claims. (Cl. 229-45.) A container suitable for ice cream, milk, and the like has a fin-and-ear type closure.

2,512,414. Method of making cigarette papers. Richard N. Booth, Wembley, England. Filed Aug. 22, 1946. 6 claims. (Cl. 164-71.) Machine for forming finished cigarette papers from a strip of the paper.

2,512,539. Easy packing container. John R. Belsinger, assignor to Belsinger, Inc., Atlanta, Ala. Filed Oct. 19, 1948. 1 claim. (Cl. 229-6.)

2,512,546. Show stand display container. Allan Hurndell, assignor of one half to Robertson & Woodcock Limited, London, England. Filed Oct. 4, 1946. 3 claims. (Cl. 206-44.11.)

2,512,577. Envelope or jacket for the safe storage of phonograph records. Albert J. Franck, Richmond Hill, N. Y., assignor of one half to Richard W. Nagel, Detroit, Mich. Filed Nov. 21, 1946. 1 claim. (Cl. 229-68.)

2,512,580. Slow-setting bituminous emulsions. Paul E. McCoy, assignor to Stancal Asphalt & Bitumuls Company, San Francisco, Calif. Filed June 14, 1948. 9 claims. (Cl. 252-311.5.) The emulsion contains 1 to 3% of sulphonated tall oil.

June 27, 1950

2,512,602. Container. John G. Bell, assignor to Morris Paper Mills, Chicago, Ill. Filed Feb, 19, 1947. 13 claims. (Cl. 229-5.5.) A multiwall cardboard container for ice cream and other food products.

2,512,648. Press roll assembly. Lloyd Hornbostel, assignor to Beloit Iron Works, Beloit, Wis. Filed May 5, 1945. 2 claims. (Cl. 92-49.) A fluid pressure-controlled horizontal press assembly.

2,512,797. Filter cartridge. Frank B. Harvuot, assignor to Bowser, Inc., Fort Wayne, Ind. Filed Sept. 28, 1945. 2 claims. (Cl. 210-619.) A laminated filter cartridge is constructed of alternate convolute layers of phenol-formaldehyde resin-impregnated paper and crepe wadding.

2,512,900. Paper winding machine sealing mechanism. George M. Kwitek, assignor of one half to Edwin M. Kwitek and one fourth to Reuben J. Pech, all of Green Bay, Wis. Filed April 21, 1948. 12 claims. (Cl. 242-56.) The machine is equipped with means for sealing both ends of a severed web immediately after its severance.

2,512,963. Paperboard serving tray. Walter L. Peiker, assignor to Continental Paper Products Company, Denver, Colo. Filed Oct. 11, 1948. 1 claim. (Cl. 229-28.)

2,512,991. Cellular carton. William H. Allen, assignor to United Board & Carton Corporation, New York, N. Y. Filed Jan. 20, 1948. 10 claims. (Cl. 229-28.) A cardboard egg container.

2,513,079. Collapsible box. William J. Buerger, assignor to The Dayton Folding Box Company, Harrison, Ohio. Filed Aug. 27, 1949. 4 claims. (Cl. 229-41.)

2,513,121. Coating compositions and method of making the same. Richard Tutt, Jr., and John R. Hubbard, assignors to Peter Cooper Corporation, Gowanda, N. Y. Filed Sept. 25, 1946. 6 claims. (Cl. 106-137.) In a paper-coating composition containing animal glue, the glue is protected against alkaline hydrolysis and bacterial breakdown by the establishment of a buffered condition in the clay prior to the addition of the glue.

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NATIONAL

Paper-Plastics Conference, New York State College of Forestry, Syracuse, N. Y., October 19–20, 1950.
General Chairman—E. C. Jahn, New York State College of Forestry.

Alkaline Pulping Conference, George Washington Hotel Jacksonville, Fla., October 25–27, 1950.
General Chairman—Henry P. Vranian, Chesapeake Corp., West Point, Va.
October 27, 1950—Visit to the Hudson Pulp and Paper Co. mill at Palatka, Fla., and the Pulp and Paper Laboratory, University of Florida, Gainesville, Fla.
Dinner party at Sportsman's Inn, Welaka, Fla.

Fibrous Agricultural Residues Conference, Northern Regional Research Laboratory, Peoria, Ill., November 13–14, 1950. Room reservations at Pere Marquette Hotel, Peoria, Ill.
General Chairman—S. I. Aronovsky, Northern Regional Research Laboratory.

Annual Meeting, Commodore Hotel, New York, N. Y., February 19–22, 1951.

LOCAL SECTIONS

OHIO SECTION:
November 9, 1950, Manchester Hotel, Middletown, Ohio.
December 12, 1950, Manchester Hotel, Middletown, Ohio.
January 9, 1951, Manchester Hotel, Middletown, Ohio.
February 8, 1951, Manchester Hotel, Middletown, Ohio.

PACIFIC SECTION:
November 7, 1950, Camas Inn, Camas, Wash.
February 6, 1951, Olympia, Wash.
April 13, 1951, Longview, Wash.

NEW ENGLAND SECTION:
November 17, 1950, Roger Smith Hotel, Holyoke, Mass.

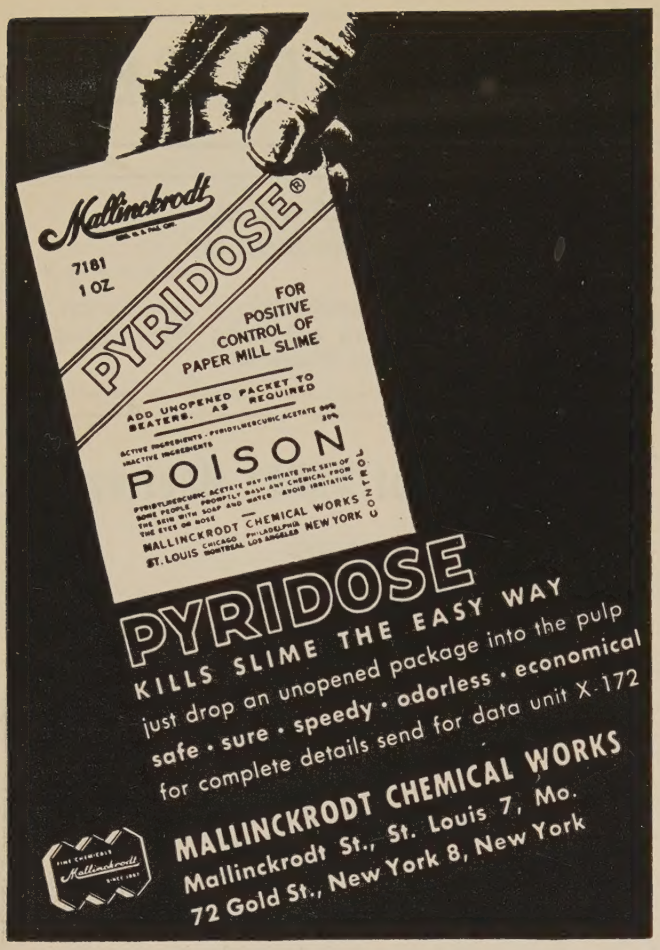
LAKE ERIE PAPERMAKERS AND CONVERTERS:
November 17, 1950, Hickory Grill, Cleveland, Ohio.
December 15, 1950, Hickory Grill, Cleveland, Ohio.
January 19, 1951, Hickory Grill, Cleveland, Ohio.

DELAWARE VALLEY SECTION:
October 26, 1950, Engineers Club, Philadelphia, Pa.
November 30, 1950, Engineers Club, Philadelphia, Pa.

CHICAGO SECTION:
November 20, 1950, Chicago Bar Association, Chicago, Ill.
December 18, 1950, Chicago Bar Association, Chicago, Ill.

METROPOLITAN GROUP, EMPIRE STATE SECTION:
November 14, 1950, Fraunces Tavern, New York, N. Y.
January 9, 1951, Fraunces Tavern, New York, N. Y.

LAKE STATES SECTION:
November 14, 1950, American Legion Clubhouse, Appleton, Wis.
December 12, 1950, American Legion Clubhouse, Appleton, Wis.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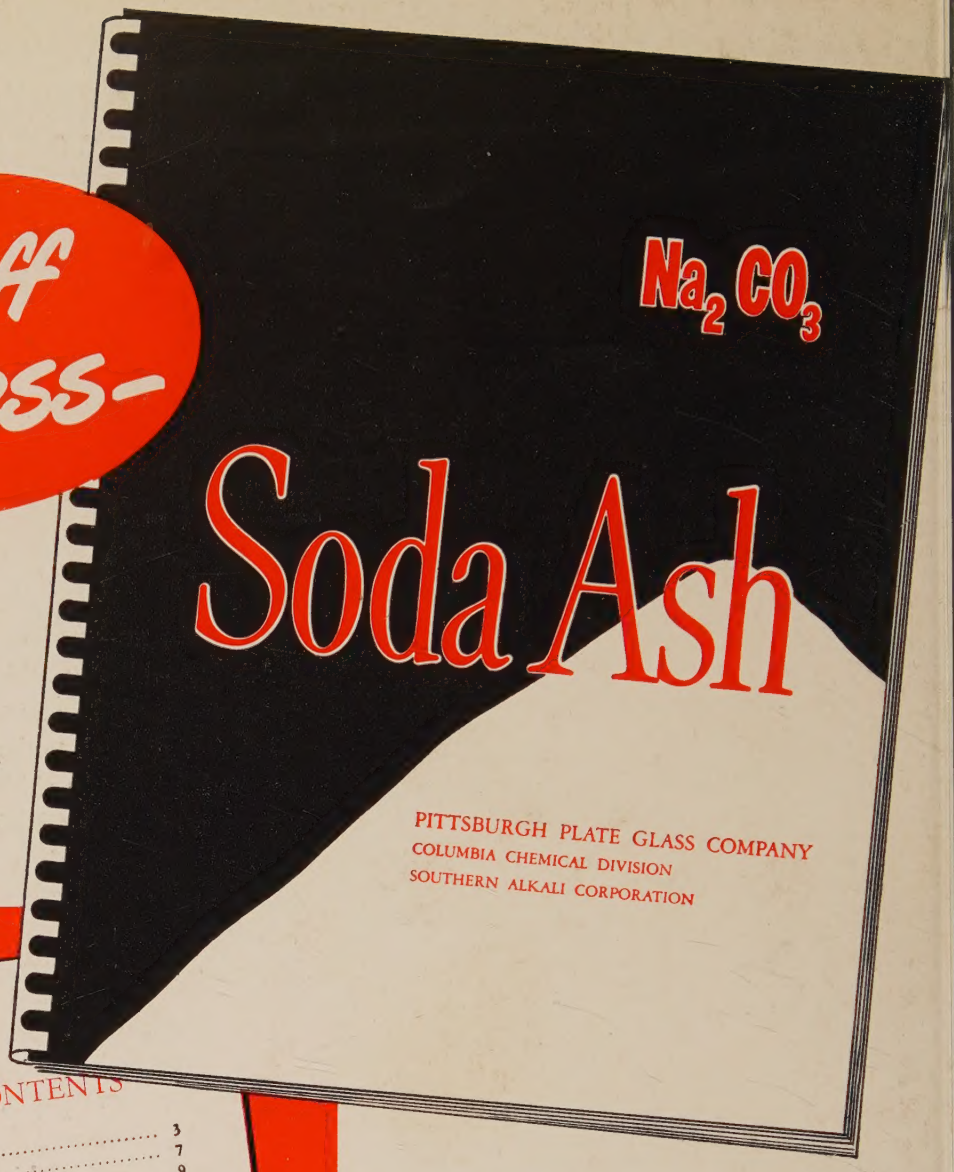

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